# AMERICAN SOCIETY
# FOR
# TESTING MATERIALS

# PROCEEDINGS
OF THE
## THIRTY-SIXTH ANNUAL MEETING

Held at Chicago, Illinois
June 26-30, 1933

VOLUME 33
PART II. TECHNICAL PAPERS

Published by
AMERICAN SOCIETY FOR TESTING MATERIALS
260 S. Broad Street, Philadelphia, Pa.

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# American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

# PROCEEDINGS, PART II

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

## SUMMARY OF PROCEEDINGS OF THE NEW YORK REGIONAL MEETING—SYMPOSIUM ON MOTOR LUBRICANTS

The fourth regional meeting of the Society was held in New York City at the Hotel New Yorker on Wednesday, March 8, 1933, in conjunction with the Spring Group Meeting of A.S.T.M. Committees. Arrangements for the regional meeting were under the auspices of the New York District Committee with the cooperation of the Metropolitan Section of the Society of Automotive Engineers. The meeting comprised a technical program, a Symposium on Motor Lubricants, to which both afternoon and evening sessions were devoted. There were approximately 350 members and guests in attendance at the meeting.

The Regional Meeting Dinner, held jointly with the Metropolitan Section of the S.A.E., was of an informal nature with but one speaker, Dr. H. C. Dickinson, Senior Scientist, U. S. Bureau of Standards, and President, Society of Automotive Engineers, who spoke on "Why not Prosper?".

The Symposium on Motor Lubricants was sponsored by the Society's Committee D-2 on Petroleum Products and Lubricants which assigned the development of the program to a special committee consisting of the following:

J. G. Detwiler (*Chairman*), Assistant Consulting Chemist, The Texas Company

R. P. Anderson, Secretary, Division of Refining, American Petroleum Institute

C. H. Baxley, Chairman, Program Committee, Metropolitan Section, Society of Automotive Engineers
H. C. Mougey, Chief Chemist, General Motors Research Laboratories
J. B. Rather, Chief Chemist, Socony-Vacuum Corporation

The symposium consisted of the following seven papers:

*Afternoon Session*

"Carbon Deposits in Gasoline Engines," by W. A. Gruse, Senior Fellow, Mellon Institute of Industrial Research
"Present Concepts of the Relation of A.S.T.M. Pour Test to Service Requirements of Oils," by J. L. McCloud, Metallurgical Chemist, Ford Motor Company
"Viscosity of Automobile Crankcase Oils as Related to Service Requirements," by E. W. Upham, Chief Metallurgist, Chrysler Corporation
"Service Changes in Crankcase Lubricating Oils," by M. A. Dietrich, Graduate Student, Ohio State University

*Evening Session*

"Oil Consumption in Motor Car Engines," by W. H. Graves, Chief Metallurgist, Packard Motor Car Company
"Factors in Engine Design Which Affect Oil Performance," by A. L. Clayden, Research Engineer, Sun Oil Company
"Aircraft Engine Lubrication," by Arthur Nutt, Vice-President in Charge of Engineering, Wright Aeronautical Corporation

The chairman of both sessions was Mr. T. C. Smith of the American Telephone and Telegraph Co., with Mr. H. C. Mougey acting as co-chairman. The papers elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on Motor Lubricants."

# CRYSTALLINE STRUCTURE IN RELATION TO FAILURE OF METALS—ESPECIALLY BY FATIGUE

## EDGAR MARBURG LECTURE[1]

BY HERBERT JOHN GOUGH, F.R.S.[2]

### FOREWORD

In accepting the signal honor conferred in the invitation to deliver the Eighth Edgar Marburg Lecture in its association with the Century of Progress Exposition, I naturally gave anxious consideration to the appropriateness of my subject. I understand that one important aim of the Exposition is to demonstrate and establish the close relation existing between scientific research and industrial development, while the Edgar Marburg Lecture was instituted to bring before the Society developments in the sciences in relation to engineering materials; it appeared, therefore, that a strong common bond existed between these aims which was not unrelated to the subject expressed in the above title. For modern industrial developments are often inseparably associated with the availability of suitable materials required for the construction of machines and structures; indeed, in many cases, urgent developments are being unduly restricted owing to the lack of such materials.[3] In an endeavor to devise new or improved materials, the metallurgist is mainly reduced to methods which can be classed as merely exploratory; he is made sharply aware that no real knowledge exists that can account, in any satisfactory manner, for the association of the observed physical properties[4] with the particular materials concerned, and, hence, he cannot attack the problem of the quest for new materials in a manner that is really scientific. Out of the darkness of this completely unsatisfactory state of affairs there shines one beacon light of future promise and hope. It lies in the fact that, whether we consider a metal or alloy merely as a solid body, as a chemical combination of various elements, or as an aggregate of crystals, it is *certain* that its properties must depend ultimately upon its inner crystalline structure as visualized by the physicist. It is established that metals consist primarily of an ordered arrangement of atomic nuclei and electrons and, therefore, the properties of metals in the bulk form as used in industry *must* be related to the atomic forces and arrangement, although that relation may not be direct and will almost certainly not be simple. Two alternative attitudes present themselves. Firstly, recognizing that the problem of the establishment of the fundamental principles of cohesion and fracture must be left—because of the highly-specialized technique involved—to the mathematical physicist, the mere engineer and metallurgist may be content to retire, temporarily, from the essential problem and await the results of the researches of his colleagues, for it appears certain that we cannot hope to build up a fundamental theory based on the results of ordinary laboratory tests made on metals in the usual form of a finely-divided polycrystalline aggregate. The alternative course became possible when large single metallic crystals were available for experimental study. By the use of metals in their simplest forms, devoid of such complicating influences as the intercrystalline boundaries, internal stress, heat treatment, cold work, etc., the physical properties of metals may be studied in relation[1] to their crystalline structure; these properties may then be compared with those of the polycrystalline aggregate and, if the results prove capable of any substantial degree of correlation, a significant advance towards the simplification of the general problems of metals will have been achieved. It will, however, be recognized that the primary fundamental problem is not attacked by such methods; nevertheless, the results should be of considerable value. For they provide data with which the correctness of a primary theory of cohesion—when available—can be examined, also, the results themselves may be of some assistance in acting as a guide to the general directions along which the work of the mathematical physicists may be usefully extended; they may also give some guidance in devising new materials of construction. It is sufficient to state that the opportunities afforded for experimental study of metallic single crystals have been pursued vigorously during the past ten years and I propose to give a brief account of that section of the work concerned principally with the characteristics of deformation and fracture under mechanical forces.[1] In spite of the progress made, a very wide field remains for future research, but there is little doubt that this is a field of promise. Already it is apparent that the deformation characteristics of metals under such diverse straining actions as static, impulsive and repeated stressing are essentially similar and that the behavior of metals during industrial operations such as rolling, drawing, extrusion, etc., can be correlated with the crystalline structure and the deformation forces.

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[1] Read on June 27, 1933, before the Annual Meeting of the American Society for Testing Materials at a Joint Session with Section M (Engineering) of the American Association for the Advancement of Science, Chicago, Ill.

[2] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

[3] The engine and structure of the all-metal high-speed airplane may be cited as typical cases where notable advances in engineering materials have enabled great progress to be achieved; the need for the employment of still higher pressures and temperatures, as in boiler plant and the gas turbine, is an example of a field where intensive research aimed at the development of new alloys may open up wide possibilities.

[4] The testing engineer is fully aware that the usual form of specification tests which he employs is designed much more with a view to ensure *uniformity of product* than to disclose the existence of properties which will be demanded of the material under service conditions; an important requirement of such tests is often the maximum simplicity of operation, while the test results can neither be correlated with service behavior nor simply interpreted. The real suitability of the material is largely determined by experience, often of a costly nature.

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[1] One indication of the advance effected lies in stress computation. In the method used by engineers for design purposes, the material is regarded as homogeneous and isotropic and average stresses only are computed; by the use of single crystals the deformation and other characteristics may be related to the true stress components in any desired crystallographic direction.

From such considerations, it is evident that the failure of metals in relation to the crystalline structure[2] forms a subject of mutual interest to the chemist, engineer, metallurgist and physicist; I concluded that, as such, it might not be inconsistent with the aims of the Society and of the Exposition.

## I. GENERAL DISCUSSION OF THE BASIC PROBLEM OF FAILURE

The study of the failure of metals in relation to crystalline structure having been conducted mainly along experimental lines, the results obtained are most usefully discussed in connection with the experiments concerned and this method will be adopted generally in this lecture. But, before embarking on these detailed aspects, some consideration may, perhaps, be usefully given to a very brief general discussion of the basic problem of the failure of metals, as it is essential that the main problem should always be kept prominently in view: *this basic problem is undoubtedly the property and nature of the cohesion of solids.* Although this is apparently a statement of a self-obvious fact it is often quite easy, in the discussion of particular problems, to ignore the basic problem, sometimes with very curious results. As a concrete example of present interest we may consider what is known as the "internal flaw" hypothesis, which has been advanced as a means of reconciling the difference[3] between the theoretical and experimental strengths of solids and which received support from the experimental work of Griffith **(81,82)**[1] Joffé **(85)** and others. Fundamentally, the mathematical application of the consequences of flaws to this problem is merely irrelevant. For the whole conception of stress concentration by flaws is founded upon the mathematical theory of elasticity, which theory is applicable only in so far as the material under consideration may be considered to be homogeneous and to obey Hooke's law. But the ultimate cause of cohesion is inter-atomic attraction and as we narrow down our investigation to an inquiry why the failure of *this* atom bond causes *that* atom bond to be overstrained and ruptured, the theory of elasticity, founded as it must be on statistical laws, becomes quite inapplicable to the problem. It is not, of course, intended to suggest that the flaw hypothesis is valueless; but that, in a fundamental consideration of cohesion, it would constitute a problem in itself rather than an explanation of a previously existing problem.

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[1] Of great importance is the work on the thermal, magnetic, and electrical characteristics of metal crystals, but these are excluded from present consideration.

[2] In discussing some aspects of the problem, in what follows, space considerations will not permit me to refer to all those investigators whose work has assisted progress in this field of inquiry; general acknowledgment is hereby rendered to those not specifically mentioned.

[3] For a detailed discussion of this aspect, see the fourth Edgar Marburg Lecture on "Cohesion and Atomic Structure" by Saul Dushman, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 7 (1929).

Until, therefore, some definite conception of the true nature of cohesion can be formed, generalization on the basis of empirical laws cannot be permitted. Hooke's law and the whole theory of elasticity must be regarded as empirical and must not be applied outside the range of experimental verification.

Our schematic knowledge of the strength and elastic properties of materials is limited seriously (and almost solely) by our complete ignorance of the nature of cohesion. The properties of a perfect gas are inherent in the laws of dynamics and although the imperfections of actual gases are not all well understood, at least they can be discussed separately as variations, usually minor variations, from an ideal behavior. Even in the case of liquids, some schematic description of the more important properties is possible; for if we neglect compressibility and viscosity, the behavior of liquids is dynamically determinable; while even inclusion of consideration of these two properties only complicates the mathematical analysis without introducing any fundamental difficulty. It is however worth noting that surface tension may seriously affect the behavior of liquids and that this property is an example of cohesion and is *not* explicable dynamically. For solids, on the other hand, no ideal behavior can be specified. In the elastic range the assumption of homogeneity, isotropy (or a regular form of aelotropy) and linear elasticity is often convenient; but it has no basis upon fundamentals. In other words, we cannot say that the fundamental property of cohesion infers these properties and that modifications or limitations in practical materials are anomalous; the basic property of cohesion might well lead to quite other properties and those we have specified might be exceptional, owing their existence, possibly, to statistical effect.

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[1] The boldface numbers in parentheses refer to the papers given in the list of references appended to this paper, see p. 105.

If the form of behavior of solids in the elastic range is a matter of doubt, the normal behavior in the plastic region is a matter of pure conjecture. I intend to go into this question more fully when discussing tests on single crystals; in the meantime, I will merely point out how closely plastic distortion is associated with failure and how impossible it is to consider fracture apart from the plastic distortion that normally precedes it.

### *A. Nature of the Atom Bond:*

I have said enough both to emphasize the importance of a knowledge of the ultimate nature of cohesion and to indicate the extreme improbability that this knowledge will ever be derivable analytically from data obtained in actual tests of materials. The only hopeful method would appear to be to attempt the synthesis of the properties of materials from assumed laws of cohesion. This method would clearly be applied with greater ease to the case of the single crystal than to that of a less regular structure; but it is equally obvious that the success of the method depends primarily on the inherent probability of the law of cohesion assumed.

Many physical facts, but particularly the structure of crystalline materials, strongly suggests that the seat of the ultimate cohesive force is the atom; that the forces of cohesion are interatomic and closely associated with the forces of chemical affinity. As evidence of this association, contrast the low cohesion of the chemically inert elements with the very high cohesion of the elements, such as carbon, which possess the maximum number of valency bonds, and consider the ease with which atoms newly detached from cohesion to other atoms enter into chemical combination. In some cases it is possible to assert almost with certainty that the major forces are electrical in nature and to identify them with the normal attraction or repulsion between particles bearing unlike or like charges; thus the alkaline halides are considered to be formed by the completion of the outer shell of the halogen atoms by the transfer of the one electron from the outer shell of the alkali element, and the molecule or the crystalline lattice is considered to be bound together by the forces between the atoms thus ionized. It is clear that ordinary electric attraction of this type cannot account entirely for the stability of the molecule; there must be some other (repulsive) force, or the two atoms would, theoretically, coalesce. Similarly the stability of the lattice requires a secondary force; for although the electric forces are both attractive and repulsive, it is obvious that their balance cannot determine the parameters of the lattice but that the electric forces either would or would not be in equilibrium depending only upon the symmetry of the lattice and not at all upon the lattice spacing.

Several workers, notably Lennard-Jones(91,92) and his collaborators, have developed the properties of ionic lattice structures along these lines. The uncertainty as to the nature of the secondary force of course constitutes a difficulty; but it has been shown that, provided the law assumed represents a repulsive force that increases rapidly as the interatomic distance is reduced, its precise form is not particularly critical.

The treatment of lattices other than those known to be ionic in structure is very much more difficult; for in this case we have in effect no real knowledge at all of the interatomic forces. It would of course be theoretically possible for *all* lattices, even those composed entirely of one kind of atom, to be ionic in structure; for individual atoms might be differentially ionized. This hypothesis has the added attraction that it allows the molecule at least partially to retain its identity in the solid state; but for other reasons this view is not considered tenable.

If all the atoms in a lattice are identical in nature, it would appear reasonable to suppose that they are identical also in their effect upon one another. If in addition the lattice forms a "homogeneous assemblage" (which means simply that no atom must be distinguishable from any other by its position relative to other atoms), then exact knowledge of the law of atomic force is no longer necessary for a preliminary investigation of the stability of the structure. In the early days of elastic theory, Cauchy developed a statistical analysis of the stability of solid matter upon just these assumptions. He showed that, provided only that the interatomic forces were directed in the line joining the atoms concerned, certain relations between the elastic constants could be deduced. While the fact of crystalline structure was not established, Cauchy's assumption of the homogeneity of the assemblage rendered his analysis inapplicable and this difference was considered to provide sufficient explanation of the experimental fact that the relations deduced were not in general satisfied; but now that single crystals of metals such as aluminum, iron, etc., are known to consist of just such homogeneous assemblages of apparently similar atoms, the non-satisfaction of the Cauchy relations would suggest strongly that it is rather the assumption as to the type of the interatomic force that is in error. The importance of this from a fundamental point of view can scarcely be over-emphasized. It is of course possible that the interatomic forces are not purely central; but the permanent asymmetry of the atom that this view would suggest is strongly at variance with the picture of the atom as otherwise developed. Born has extended the field of inquiry by postulating that the force between two atoms is affected by the proximity of other atoms; but as this method applies only to certain types of structure (ionic) it may be inapplicable with regard to metals. It may be considered that I am giving undue attention to the discussion of these Cauchy relations; but to an engineer, they do appeal as relatively solid ground from which to survey the quicksands that seem to surround the problem of cohesion.[1] Although space will not permit reference being made to all the hypotheses that have been advanced regarding the nature of cohesion, I would mention the types of cohesion assumed to exist in those substances governed by "homopolar" and "metallic" types of combination. In the homopolar type, an arrangement of atoms—individually unstable due to incomplete electron shells—is rendered stable by a process of electron sharing; diamond is an example of this class. In the metallic type of combination, modern theories regard the positive metallic ions as held in equilibrium by mutual interaction with a "gas" of electrons. Recently developed theories consider the motion of electrons as a transfer of electron waves through a periodic potential field provided by the regular ionic lattice; a process which may be likened to the passage of light waves through a medium having a periodically varying refractive index. With the introduction of the Fermi quantum statistics, the new theories have successfully solved problems in specific heats and the motion of electrons in strong fields. It is possible that these new theories may throw light on the forces of cohesion in metals which will be in better agreement with experimental values than the Cauchy relations and lead to the calculation of new relations between the elastic constants; attempts in this direction have been made by London and Heitler(**94**).

### *B. The Structure of Solids:*

The suggestion made by Laué that the use of a crystal would reveal whether X-rays and light were of the same nature, the success-

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[1] The determination of the elastic constants of metallic single crystals is of very great importance in providing data for quantitative tests of theories of cohesion. Notable work in this field has been carried out by Bridgman(**11**), Geiss(**38**), Goens(**40**) and his collaborators(**41,83**); Subrahmaniam(**134**) and Wright(**163**). The dynamical method, as used by Wright, is capable of very great accuracy and reduces the possibility of errors arising from the low values of the primitive elastic limits associated with certain crystals.

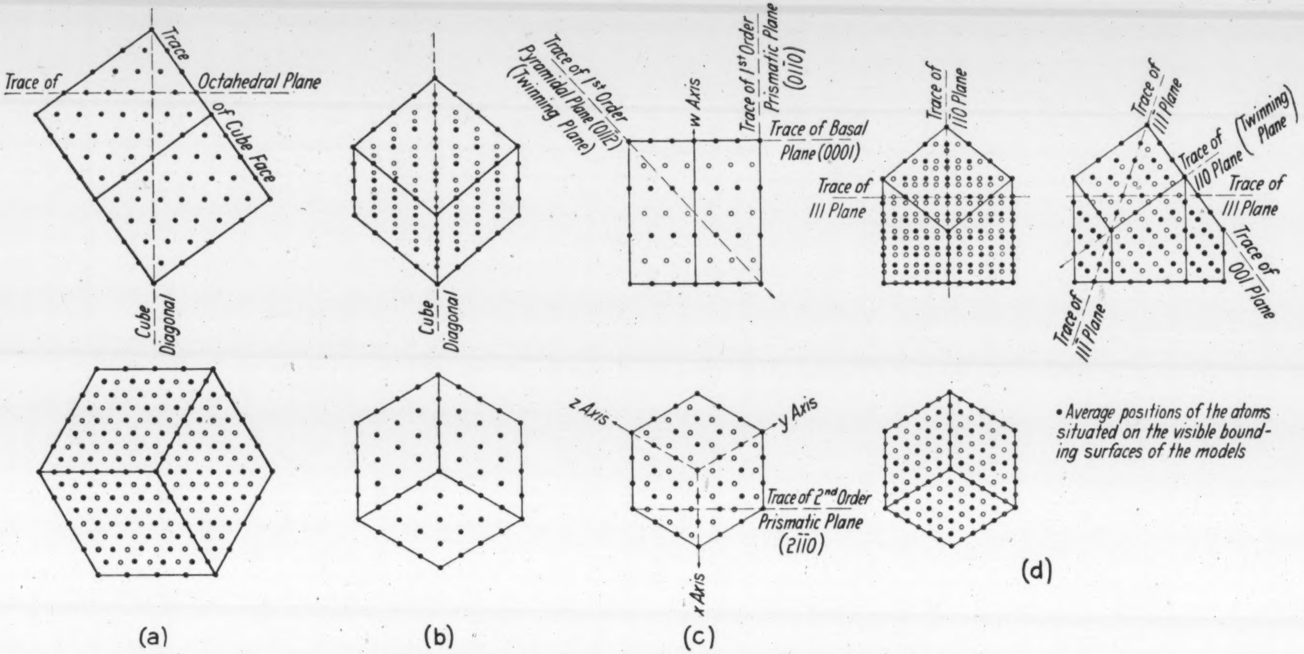
Trace of
Trace
Octahedral Plane
of Cube Face
Cube Diagonal
Cube Diagonal
Trace of 1st Order Pyramidal Plane $(01\bar{1}2)$ (Twinning Plane)
w Axis
Trace of 1st Order Prismatic Plane $(01\bar{1}0)$
Trace of Basal Plane (0001)
z Axis
y Axis
x Axis
Trace of 2nd Order Prismatic Plane $(2\bar{1}\bar{1}0)$
Trace of 110 Plane
Trace of 111 Plane
Trace of 111 Plane
Trace of 110 Plane (Twinning Plane)
Trace of 111 Plane
Trace of 001 Plane
Trace of $\bar{1}11$ Plane
• Average positions of the atoms situated on the visible bounding surfaces of the models
(a)
(b)
(c)
(d)

FIG. 1.—Diagrammatic Representation of the Structures.

(*a*) Face-centered cubic.
(*b*) Body-centered cubic.
(*c*) Close-packed hexagonal.
(*d*) Face-centered rhombohedral.

ful experiment by Friedrich and Knipping, the work of W. H. Bragg, W. L. Bragg, and others—in short, the use of X-rays in determining crystal structure—led to an enormous and very rapid expansion of our knowledge of the atomic structure of matter.[1] Previously, knowledge was limited to a theoretical description of the ways in which material points or particles could be disposed to form a homogeneous body and to reasonable inferences on the basis of this theory as to the structure of those crystals that could be grown to exhibit their symmetry in their external form.[2] Consideration of symmetry operations alone is sufficient to show that any type of regular structure in three dimensions must be constructed on one of fourteen types of lattice, the lattice points defining points of complete identity of the structure; while, again, symmetry restricts the number of point-groups that can be built on these lattices to 230 possible types. Figure 1 shows diagrammatic representations of four lattice structures of metals. The cubic structures are of high symmetry and form examples of a "simple" lattice, in which the atoms form a "homogeneous assemblage," as previously defined. The close-packed hexagonal and face-centred rhombohedral structures, however, are of a complex nature, the atoms being divided into one or more sets distinguishable positionally one from the other. As an alternative to these regular structures, it is possible to conceive an entirely random distribution of points, characterized only by a statistically uniform space density.

Now, analysis of materials by X-rays has shown that regularity of structure is a feature of a very large proportion of all solids:[3] it may even occur in some liquids. Metals in the normal state usually consist of an aggregate of very small crystals of random orientation, this structure being due to crystallization from the liquid proceeding simultaneously from a number of different nuclei (the acicular structure of chill-cast bars illustrates the process); by very slow uniform cooling under controlled conditions it is possible to restrict the number of nuclei and to grow very large crystals. The intercrystalline boundaries are usually quite irregular in shape; but no method has yet been devised for the investigation of the atomic structure in the neighborhood of the boundary. Two extreme views are possible; first, that each crystal abuts on to its neighbors without any deformation of its lattice; second, that each pair of crystals is separated by a layer of "amorphous" material. Reflection shows however that neither view is really tenable. The forces on an atom near the boundary are composed of two sets, each tending to bring the atom into the lattice of one of the crystals bordering the boundary. The final probable position of the atom would then appear to be in the nature of a compromise, thus leading to a form of gradual (gradual in the sense of being continuous) transition from the orientation of one crystal to that of its neighbor. Such a "curved" lattice represents a possibility intermediate between the two extremes mentioned: at one time, these three conceptions appeared to be the only possibilities. But the results of X-ray analysis of cold-worked structures led me to suggest (66) a new and alternative hypothesis, namely, that the transition was effected by a number of very small crystallites of slightly differing orientation, the structure of each being appreciably free from lattice distortion but having strained or ruptured atomic bonds at the junctions; further reference will be made to this hypothesis.[1] It is, therefore, no longer *necessary* to invoke the existence of an amorphous state of metals to explain the structure of the intercrystalline boundary. With regard to the existence of the amorphous condition in metals, it is necessary to realize that means have not yet been devised to differentiate with certainty between truly amorphous material and structure composed of extremely small crystallites. If investigation could be restricted to a sufficiently small number of atoms, in the limit, of course, the distinction would become ridiculous, but although X-ray analysis does at the moment favor an amorphous state in some glasses and leaves the matter an open question in the case of surface films[2] produced by polishing of metals, the number of atoms the beams covered is too large for this evidence to be regarded as definite one way or the other. If the amorphous state exists in solids, it is probably to be regarded as the state of a supercooled liquid and the production of material in such a state is presumably to be ascribed to causes similar to those responsible for the supercooled vapor state; it will be noticed that, on this hypothesis, the amorphous state would be a state of high internal energy, the material retaining the latent heat of melting. Personally, I consider that, until definite evidence is obtained of the existence of the amorphous state in metals, this hypothesis requires no further consideration. During the period when knowledge of the structure of metals was based on visual observation or other indirect methods, the amorphous theory was suggested as a possible explanation of certain observed phenomena exhibited by metals; it certainly stimulated vigorous and useful discussion of these phenomena. But the wider vision conferred by the use of X-rays and the studies of metallic single crystals has created an entirely new outlook as quantitative data are now available to replace previous mere surmises. Therefore, in the present attempt to build up a modern view of the failure of materials based on the behavior of metals in their simplest forms, it appears to me to be entirely unnecessary to invoke the aid of the older hypotheses.

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[1] See works by Bragg(7,10), and Wyckoff(164); a conception of the value of applied X-rays in modern industry will be gained from the following references: Bragg(8), Clark(16), and Pullin(110).

[2] The results obtained by crystallographers, without the use of X-rays, were truly remarkable; in this connection, the work of Tutton(148) will never be forgotten.

[3] Even such materials as asbestos, stretched rubber and worked grease have been found(16) to exhibit a crystal structure.

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[1] The close resemblance between the conditions at the intercrystalline boundaries of a polycrystalline aggregate and the junctions of the crystallites into which a single crystal is broken up lends considerable support to the theory. For if a cold-worked aggregate is recrystallized, the new grains usually have their origin at the original boundaries; if a cold-worked single crystal is recrystallized, the orientation of the new grains is random, bearing no relation to that of the original crystal.

[2] The use by Thompson (147) of the Electron Diffraction method in investigating surface layers offers great promise in this direction as layers of thicknesses equal to a few tens of atoms only can be examined; the results already obtained, however, are somewhat inconclusive as diffraction patterns could be obtained neither from a polished metallic surface nor from an etched metal surface.

Before passing to the detailed examination of the results of tests on single crystals, the degree of perfect symmetry obtaining in these crystals demands brief discussion. Deformation characteristics, as we shall see, suggest forcibly that the ordinary metallic single crystal does not possess perfect symmetry; a very difficult problem is encountered when we endeavor to ascertain whether these characteristics really do imply discontinuity of structure or merely result from the inherent nature of the forces of cohesion of which we possess such extremely limited knowledge. Reference has already been made to the "internal flaw" hypothesis which appeared to offer a not unreasonable explanation of the difference between the practical and the estimated theoretical strengths of solids; in fact, Griffith(**81, 82**) and Joffé(**85**) demonstrated that even the full theoretical strength might be attainable under special conditions. While objection to the hypothesis as a *fundamental* concept could still be lodged, disbelief in the existence of flaws as a practical explanation was difficult to substantiate. It could be argued that (on ordinary elastic theory) the existence of flaws implied a total energy content above the minimum; but there was no sound reason why metals in the ordinary polycrystalline form *should* have minimum energy. When, however, it was proved that failure (particularly fatigue failure) normally commenced inside the crystal grains and when, further, it was shown that single grains, grown in such a manner that the internal energy should be as low as possible, were perhaps even weaker than the polycrystalline material, it became necessary to devise some more satisfying explanation of the relation of the flaws to the crystalline structure. Such an explanation[1] has recently been advanced by

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[1] This should be regarded as an extension of the earlier theory of Smekal; see full discussion by Dushman(**20**).

Zwicky(171,173,174,175) and experimental evidence in its support has been adduced by Goetz(42,43,44,45,46,47). Briefly, Zwicky concludes that, in addition to the symmetry forces producing the true lattice structure, there is a secondary system by which the primary structure is broken up into smaller units, of which the "edges" constitute partial flaws. It is too early yet to judge the value of Zwicky's work, but I should perhaps mention that in addition to the experiments of Goetz, the X-ray results obtained by W. L. Bragg and others on extinction-coefficients tend also to support the theory. The behavior of single crystals under straining forces is also *consistent* with the mosaic structure hypothesis.

Nevertheless, it must be pointed out the assumed discrepancy between the theoretical and actual strengths of solids is not even necessarily *applicable* to the ductile metals in which we are primarily interested. For the calculated theoretical strengths assume that, immediately prior to fracture, *all* the atom bonds are uniformly strained, whereas, in ductile metal crystals, failure is usually preceded by considerable plastic deformation; this deformation occurs by shearing movements or by twinning, neither of which movements is in any way representative of the complete separation envisaged in the theoretical estimate of strength. As will be seen, both slip and twinning at least partially destroy the lattice and probably provide the minute cracks that ultimately lead to complete failure. Our fundamental problem therefore becomes the strength of metals under shearing forces and not under those forces producing failure by the direct separation of planes of atoms; it remains, however, as a problem of cohesion. The suggestion is therefore advanced that we are not yet in a position to discuss whether the observed strength of ductile metals *is* abnormal. This position can only be reached when, the true nature and extent of the inter-atomic forces having been determined, the theoretical resistance of a perfect crystal to *shearing forces* can be calculated.

## II. METHODS OF PREPARATION OF SINGLE METALLIC CRYSTALS

Although metals do not normally occur in the form of single crystals, success has attended the artificial production of large crystals using each of the three methods which, theoretically, are capable of producing the desired result. The first method is the production of the crystal from the vapor phase, the second from the liquid phase, the third by the conversion of solid metal, in the usual form of a finely-divided aggregate, into a single crystal. Using these general methods, at least nine separate techniques(14) have been developed for making large single metallic crystals. Techniques due to Koref and Van Arkel have been used to prepare single crystals of iron, hafnium, molybdenum, tantalum, thorium, titanium and zirconium from the liquid phase. Bridgman, Czochralski, Davey, Goetz, Gomperz, Kapitza, Mark Polanyi and Schmid, Obreimow and Schubnikow, Rosbaud and Schmid, and Tammann have devised methods by which, from the liquid phase, single crystals of silver, aluminum, gold, bismuth, cadmium, copper, lead, antimony, tin, tellurium, zinc and many alloys have been prepared. The methods of converting polycrystalline aggregates into single crystals, due principally to Carpenter and Elam, Edwards and Pfeil, and Goucher, have been used to make single crystals of aluminum, iron, molybdenum, tungsten, also single crystals of various solid solution alloys.

In general the methods of production employed do not enable any desired crystal orientation to be produced at will, although several special techniques have been successfully devised to attain this end; this aspect, however, is not of importance in the present connection, as the accumulation of test data has enabled simple laws of deformation, independent of orientation, to be deduced. Of more importance is the inquiry as to the extent by which the degree of perfection of the resulting crystal is affected by the method of preparation. At the present time there is evidence that certain methods possess relative advantages; a sufficiently comprehensive study of crystals of the same type but made by various methods has, however, not yet been carried out that would enable general conclusions to be drawn: this aspect of the subject is deserving of future experimental attention.

## III. CRYSTAL STRUCTURE

The essential feature of the crystalline structure of metals, as revealed by X-rays, is the periodic arrangement of the mean positions of the atoms. Due to this we can consider, as a general conception, that the centers of symmetry groups of atoms are situated at the points of a lattice whose dimensions and form vary from metal to metal but constitute one of a fixed number of possible arrangements. A simple lattice is formed if three vectors, $a$, $b$ and $c$, not lying in the same plane, also the three corresponding reversed vectors, $-a$, $-b$, and $-c$, are projected from any origin $O$, the arrangement being continuously repeated from the six end points thus obtained. The directions of these three vectors are called the "primitive directions" while the actual values of $a$, $b$ and $c$ are known as the "primitive translations." A structure built on a parallel lattice, which is *not* simple, may be produced if the same primitive directions and translations are applied not to the one origin $0$, but to a series of origins, $0_1$, $0_2$, etc.; such a parallel lattice may also be regarded as a set of interpenetrating congruent simple lattices in which corresponding vectorial directions remain parallel. From symmetry operations, a maximum of 230 crystal groups are obtained: every crystalline form must conform to one of these groups. It happens that the majority of metals which are of importance as materials of construction conform to one of three lattice systems: the body-centered cubic, the face-centered cubic or the close-packed hexagonal; the first two of these are *simple* lattices while the third is a *parallel* lattice of an elementary type. In the body-centered cubic lattice, the three primitive directions[1] correspond to three of the diagonals of the elementary cube, while the three primitive translations are equal to each other, having a value of $\frac{\sqrt{3}}{2}$ times the side of the elementary cube. The face-centered cubic structure has, as primitive directions, the three intersecting diagonals of the cube faces, while the primitive translations are each equal to $\frac{\sqrt{2}}{2}$ times the length of the cube edge. The close-packed hexagonal system is made up of two interpenetrating congruent simple lattices: each of these consists of two equal primitive translations intersecting at an angle of 60 deg., while the third and unequal axis is perpendicular to both: the ratio of the length of the third axis to that of either of the remaining two is known as the "axial ratio." It may be mentioned that even a *simple* hexagonal close-packed lattice only corresponds, geometrically, to a close packing of *spheres* for the particular value of the axial ratio equal to $1.6\dot{3}$; the actual value for zinc, for example, is 1.86.

A direct consequence of the geometrical regularity of the lattice system of a crystal is that all the lattice points can be considered as lying on a series of parallel planes of any one type: in any such series, the atomic density (lattice points per unit of superficial area) will be equal and the average distance between adjacent planes will also be equal (in a simple system such as the cubic, the planes will be quite regularly spaced). The *planar* atomic density will be inversely proportional to the average interplanar distance; similarly, the *linear* atomic density (lattice points per unit length) in directions contained by any plane will not, of course, be a constant quantity but will be dependent upon the lattice structure and on the relative orientation of the direction in question with respect to the lattice structure. A crystal must therefore be regarded, essentially, as an anisotropic solid, possessing directional properties; it is not surprising, therefore, to find that the distortion of metallic crystals is related not only to planes of a certain type but also to certain directions in those planes. To distinguish such planes and directions it is convenient to make use of the well-known Millerian system of indices, distinction being effected by reserving to planes the appropriate indices contained in a "round" bracket, thus (111), those referring to directions[1] being contained in a square bracket, thus [110]. The Millerian indices define completely any plane or direction in relation to axes defined by the crystalline structure; in actual experimental work, however, we require to refer these planes and directions to arbitrary axes. The spherical coordinates of the planes and of the directions relative to these arbitrary axes are therefore employed.[2]

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[1] The method adopted here differs from that commonly used in many engineering and metallurgical publications where the cubic system is usually referred to the cube edges: the latter method is not, however, correct (although it is, perhaps, more convenient) because, by its use, the body-centered cubic and face-centered cubic arrangements appear to be *parallel* lattices whereas, in fact, they are *simple*.

Having thus glanced at what may be termed the "geometrical" aspects of crystal structure, the ideal way of treating the subject in detail would be to discuss the observed characteristics and failure of metal crystals in relation to the properties theoretically pertaining to such crystals. I cannot do this as the fundamental theory is not available. As a result, certain phenomena will be encountered which, for all we know, may, in fact, be consistent with the properties of crystals of perfectly regular structure but which, owing to our ignorance and tendency to accept preconceived ideas of the interatomic forces, appear to be abnormal. I propose, therefore, to adopt a method which may invite serious criticism but which I have personally found useful in formulating my own ideas, namely, to consider the properties which a crystal should possess according to one of the older theories and compare the behavior of actual crystals with these assumed properties. May I make it quite clear at the outset that I do not believe in the theory that I shall set up to act as a sort of coconut shy, but the method has led me to the view that the newer theories of metals—in which the interatomic forces are periodic—may render their observed behavior entirely comprehensible. But my chief reason for employing this very questionable method arises from the hope that, by its use, many who are only *generally* interested in the whole subject, will perceive that many commonly observed phenomena of metals—so common that they are accepted without question—do not admit of a simple explanation; if, as engineers and metallurgists, we can arrive at this conclusion, it will be brought home to us at once that the methods of modern physics are being employed on *our* problems. Such appreciation of the work of the mathematical physicists—even though we shall often be unable to follow the method or even the form in which the result is expressed—cannot but be stimulating and helpful. The alternative method is to be content with out-of-date physical theories and, in the attempt to reconcile these with our own observations, invoke even more improbable "explanatory theories." I regard the "flaw" hypothesis (in its original crudity) as one such unsatisfactory effort at compromise. With less sure conviction, the "mosaic structure" theory—as a *static* or *fixed* (with relation to the primary lattice) structure—is similarly regarded, although it is freely admitted that much experimental evidence has been adduced in its favor. But if the secondary structure can be regarded as moving periodically through the lattice —I do not know if Smekal, Zwicky and Goetz are prepared to accept this interpretation—it appears to me that the conception is neither inconsistent with the modern ideas of "wave mechanics" when applied to solids nor, necessarily, with the observed characteristics of metals.

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[1] These indices really specify the *plane* to which the given direction is normal: no difficulty is involved in simple lattice systems, but care must be exercised in some complex systems in order to avoid confusion if simple indices are employed for convenience.

[2] For notes of a convenient reference system for use in experimental work on single crystals, the reader is invited to consult a paper(67) by the author. The use of the stereographic net is also described.

After this digression, the probable properties of our hypothetical crystal may be formulated. The lattice is a mechanical system in a state of stable and static equilibrium, each atom being regarded as a detached rigid structure, the balanced inter-atomic forces of attraction and repulsion being merely dependent on mass and inter-atomic distances; relative atomic displacements from the equilibrium position invoke restoring forces. Referring to such a crystal as an "ideal" crystal, its form represents essentially one of stability and minimum potential energy; also, marked rigidity and elasticity would be expected as primary properties. Under the action of applied straining forces, we might encounter movement from the original structure to an alternative position of stability and equal potential energy, but the initial and final states would be indistinguishable by X-ray methods; a truly translatory movement might be responsible. At some higher load, or due to another type of straining action, the applied forces would, conceivably, be sufficient to overcome the cohesion of the lattice structure and lead to complete fracture by separation of planes of atoms. But the phenomena of strain-hardening, etc., associated with plasticity of metals—observed in experiments to be described later—are foreign to such a crystal. Also, failure by repeated cycles of stress or "fatigue," one of the most important aspects of engineering materials, should not be possible in the ideal crystal: it will be shown that fatigue and plastic deformation are related phenomena in actual metals. It may be suggested that the discrepancy between the behavior of the practical crystal and the suggested characteristics of the ideal crystal may be accounted for by the relative impurity of the former; this explanation, however, is insufficient, for experience has shown that the plastic behavior becomes more pronounced with an increasing degree of purity of the single crystal. One possible flaw in the argument, however, must not be overlooked. In the ideal crystal we have considered a lattice which extends indefinitely in all directions, whereas our actual crystals, being of a finite size, possess a free surface. Obviously the conditions of inter-atomic equilibrium must be affected at such a surface and a very considerable amount of evidence has been afforded, principally by Joffé(85) and his collaborators, which seeks to explain the main problem by imperfections at, and due to the existence of, the free surface. Although the general body of present-day informed opinion favors causes which are not *confined* to the free surface, the possible consequences of the latter, in relation to our problem, must never be lost sight of.

The detailed and statistical study of the mechanism of the plastic distortion of metals by slip has all been carried out within the last decade, but the literature of the subject is already very voluminous. This study may be considered as having been investigated in two main directions. In one, the geometry and other aspects of distortion have received attention in experiments which involve considerable changes of shape of the specimens used; in the other group—made by the author and his colleagues and directed principally to the study of fatigue phenomena—the conditions of the experiments have been purposely chosen so that fracture is produced with the least possible change of the external dimensions and shape of the specimens. It is indeed significant and satisfactory to find that, at the present period of review, the observations obtained in the two fields of research appear definitely to converge on the same fundamental causes of failure; the same basic problems are involved and, in both cases, a correct understanding of slip phenomena appears to be of profound importance. Before reviewing the work on the fatigue of single and large crystal specimens, an attempt will be made to summarize some of the characteristics exhibited by metallic crystals when deformed statically. The space at my disposal makes it quite impossible to give a complete summary of the vast amount of valuable research work which has been completed, chiefly, by American, British, German, Russian and Japanese investigators: I must be content merely to choose a few typical examples of experiments to demonstrate some characteristics of metallic crystals which are of particular interest in connection with the general purpose of the present paper. Also, as no attempt will be made to follow any chronological order, it may be mentioned that three principal and independent lines of research were commenced about the same time. The first accurate study of the deformation of single metallic crystals is described in a paper by Mark, Polanyi and Schmid(96) published in December, 1922. The first of the studies of Taylor and Elam was published(140) in 1923. The first series of fatigue experiments on single crystals was published by Gough, Hanson and Wright(59) in 1924.

## IV. The Distortion of Single Metallic Crystals Under Simple Static Stressing Systems

In general, two methods have been adopted in studying the distortion of single crystals under static forces. The more exact method is that devised by Taylor and Elam(140), in which the relation between the distortion and the crystal axes is determined. Reference marks are placed initially on the specimen and, by purely analytical means, the directions in the crystal which remain unchanged in length when the specimen is subjected to various stages of a uniform distortion are determined. In the general case, these directions form an unstretched cone of the second degree. By means of X-ray determinations of the crystal axes, the cone can be related to the crystalline structure. Now, if a material deforms by a simple shear, the unextended cone degenerates into two planes, the direction of slip being contained by one plane and being perpendicular to the intersection of the planes. In aluminum, Taylor and Elam found that the unextended cone *did* degenerate into two planes, one of which bore a strict and constant relation to the crystallographic axes, while the other bore no such relation, its identity varying with the amount of strain. In this way it was demonstrated analytically, for the first time, that the normal process of plastic distortion of a single crystal was by one of shear on certain crystal planes along certain crystallographic directions contained in these planes; later, the same method assisted to establish the law that, of the various crystallographically equivalent slip planes and slip directions, the operative[1] plane and direction was that subjected to the highest value of shear stress when resolved along the slip plane and in the slip direction. In England, this law is usually referred to as the "maximum resolved shear stress" criterion.

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[1] The term "operative" will be employed to denote the plane on which slip occurs during the period under discussion.

The second method of determining the mechanism of distortion lies in correlating the results of observations of the changes in external shape of the crystal, of the inclinations of surface markings which appear on the specimen and of the changes in relative orientation of the crystallographic and specimen axes. The nature of the distortion is then determined by implicitly *assuming* that the process of deformation is one of shear on crystal planes and that the observed surface markings are the visible external consequences of the distortion: the identity of the slip plane and direction can then be deduced. It is the assumption made in the second method, first used by Mark, Polanyi and Schmid(96), that differentiates it sharply from the first; in effect, whereas, in the second method, the mechanism of distortion is first assumed and then put to the test, the analytical method[1] requires no assumption. Both methods have contributed enormously to the study of distortion.

Resulting from the work of many researchers, it has been shown that the distortion of single metallic crystals occurs by one, or both, of two distinct mechanisms: by slip or by twinning. The mechanism of slip is equivalent, geometrically, to one of shearing[2] on certain planes in directions lying in those planes; the first, and simpler, part of the problem is to determine the relation, if any, which exists between these slip planes and directions and the crystal structure. It has now been established, satisfactorily, for many metallic crystals that the slip direction always coincides with a crystallographic direction of maximum linear atomic density; to this criterion no exception has as yet been encountered. With regard to the slip plane, this has been found, *in general*, to be parallel to those crystallographic planes, containing the slip direction, on which the atomic density is equal to, or exceeds, that of any other plane containing the same slip direction. At least one exception—that of alpha iron, conforming to the body-centered cubic lattice—to this general rule has, however, been definitely established. In two metals at least, namely, bismuth and antimony (conforming to the face-centered rhombohedral lattice) the process of plastic deformation by *slip* has not yet been definitely observed.

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[1] This method cannot, of course, be used with certainty for investigating distortions which are not uniform and of a simple character; the distortion of crystals which deform by a combination of slip and twinning, for example, could not be accurately determined in this way. Such complex cases are best dealt with by a method involving a correlation of X-ray analysis and careful microscopical examination of the changes in microstructure resulting from the distortion: the author has developed a special technique(65) suitable for the study of single crystals under fatigue stressing.

[2] This must be regarded only as the gross or average effect, the actual local distortion involved being much more complex; this aspect will be referred to later.

Two adjacent parts of a crystal are said to be in twin relation when these parts are symmetrical, crystallographically, with respect to each other and to a common crystallographic plane. This common plane, which often does *not* form the separating boundary between

TABLE I.—PLANES AND DIRECTIONS OF SLIP, ALSO TWINNING PLANES, IN METALS, WHICH HAVE BEEN ESTABLISHED EXPERIMENTALLY.

| Lattice Structure | Metal | Slip Plane | Slip Direction | Twinning Plane | Plane of Greatest Atomic Density | Direction of Greatest Atomic Density | References to Some of the Principal Memoirs Describing the Mechanism of Distortion of Metals, by Slipping and/or Twinning. |
|---|---|---|---|---|---|---|---|
| Face-centered cubic.... | Aluminum<br>Copper<br>Silver<br>Gold<br>Copper-Zinc<br>Copper-Aluminum<br>Aluminum-Copper<br>Aluminum-Zinc<br>Gold-Silver<br>Copper-Gold | (111) | [110] | (111)<br>(111)<br>(111)<br>(111)<br>Probably (111) | (111) type | [110] type | Elam(21,22,23,24,25)<br>Göler and Sachs(49,50)<br>Gough and Cox(73)<br>Gough, Hanson and Wright(59,62)<br>Gough and Sopwith(78)<br>Karnop and Sachs (86,87,88)<br>Phillips(107)<br>Sachs and Shoji(114)<br>Sachs and Weerts(115)<br>Taylor(137)<br>Taylor and Elam (140,141)<br>Taylor and Farren(143)<br>Yamaguchi(167,168,169, 170) |
| Body-centered cubic...... | Copper-Zinc<br>Alpha Iron<br>Tungsten | (110)?<br>(110)?(112)?<br>(123)<br>(112) | [111] | (112) | (110) type | [111] type | Fahrenhorst and Schmid(32)<br>Göler and Sachs(48)<br>Goucher(52,53,54)<br>Gough(65)<br>Mathewson and Edmonds(99)<br>Mügge(101)<br>O'Neil(104)<br>Pfeil(106,105)<br>Smith, Dee and Young<br>Taylor(139)<br>Taylor and Elam(142) |
| Close-packed hexagonal..... | Cadmium<br>Zinc<br>Magnesium<br>Zinc-Cadmium<br>Zinc-Tin<br>Beryllium | (0001) | $[2\bar{1}10]$ | $(10\bar{1}2)$ type | (0001) | $[2\bar{1}\bar{1}0]$ type | Boas and Schmid(5)<br>Goens and Schmid(41)<br>Gough and Cox(68,69)<br>Mark, Polanyi and Schmid(96)<br>Mathewson(98)<br>Mathewson and Phillips(100)<br>Rosbaud and Schmid(111)<br>Schiebold and Siebel(116)<br>Schmid (118,119,120,121)<br>Schmid and Wassermann(124) |
| Rhombohedral | Bismuth<br>Antimony<br>Tellurium | ........<br>$(10\bar{1}0)$? | ......<br>$[10\bar{1}0]$ | (011) type | $(01\bar{1})$ type<br>(0001) | $[01\bar{1}]$type<br>$[10\bar{1}0]$type | Georgieff and Schmid(39)<br>Gough and Cox(70,72)<br>Schmid and Wassermann(123)<br>Wassermann(150) |
| Tetragonal..... | Tin | (100) and (110) | [001] then [101] | (331) | (100) then (110) | [001] then [100],[111], [101] | Mark and Polanyi(95) |

the component parts of the crystal, is termed the *twinning* plane. Usually, the orientation of these parts, with respect to the twinning plane, is such that, if both parts were rotated through the same angle about a common axis contained by the twinning plane, a uniform common orientation would result; in most metals, the parent and the twinned structures constitute "mirror images" about the twinning plane. It is highly probable that the mechanical formation of a "twin band" represents the summation of a number of atomic translations and rotations, the movement of each individual atom, with respect to its neighbors, being less than an inter-atomic distance. Deformation by mechanical twinning and by slip are thus essentially different mechanisms of distortion,[1] producing structures which are capable of differentiation by the use of X-ray analysis and etching methods. Considerable total deformation can be produced, particularly in certain metals, by mechanical twinning;[2] in some cases, this is a direct result of the twinning, in others, for example, zinc, considerable opportunity for further distortion is provided by *slip* within the twinned structure. Some metals twin readily, others only under exceptional circumstances; again, in some cases, slipping precedes twinning and *vice versa*. Our knowledge of the stress conditions governing twinning is not capable of generalization; in fact, within the author's knowledge, the only attempts made to study mechanical twinning in relation to the responsible stress system are those recorded (68,69,70,72) by the author and his colleague, H. L. Cox.

The present position regarding the identity of the slip plane, slip direction and twinning planes in metals is summarized in Table I, in which some data are included regarding the relative atomic density of the principal crystallographic planes and directions.

The results which are summarized in Table I offer opportunities for extremely lengthy discussion, but such cannot be attempted in the present connection, where it is proposed to concentrate on the *general* aspects of the main problem of failure; attention will, therefore, be confined to the description of a few typical characteristics.

Considering plastic deformation of single crystals by slip, the first general feature of interest is that distortion proceeds by a process in which a certain direction remains unchanged in length and that this slip direction is identified with the direction of maximum linear atomic density. Any such direction will be contained by a single infinity of planes of varying planar atomic densities. Table I shows that, *in the majority of cases* investigated, deformation also occurs by shearing on the *plane* of maximum atomic density; this general criterion holds in all cases for the face-centered cubic lattice and the hexagonal lattice; also, possibly, for the tetragonal lattice. If it had been found that plastic distortion by slip *always* proceeded by a process of simple shear on planes of maximum atomic density in the contained direction of maximum linear atomic density, this would have been a very simple and convenient criterion. But the distortion of metals conforming to the body-centered cubic lattice at once provides a definite exception to this simple mechanism; the single crystals of this type which have been studied are alpha iron, tungsten and beta brass. With regard to the latter two materials, the author considers that further investigation of the slip mechanism can usefully be made, but sufficient accurate knowledge is definitely established regarding alpha iron to make it certain that the mechanism of distortion is *not* one of simple shear on the plane of maximum atomic density. Now there are two ways of regarding the mechanism of distortion in general. It may be considered that a general law (involving only planes and directions of maximum atomic density) has been *established* to which three metals (at least) form *exceptions*. Alternatively, it may be said that, although the slip direction always coincides with the crystallographic axis of maximum linear atomic density, no general law connects the slip plane with the atomic density on that plane, except to the extent that the slip planes are always of high[1] density. But in the absence of exact knowledge of any reasons why exceptions to the supposed "law" should exist, it is dangerous to accept the first point of view. It must also be remembered that our early ideas were, probably, unduly influenced owing to the fact that intensive study of the behavior of single crystals was first given to those metals (chiefly aluminum and zinc) which were most easily prepared; it so happened that these were found to deform in the simplest manner. Having stated the present general position, we may discuss in more detail some characteristics exhibited mainly by single crystals deforming in the simplest manner, namely, on clearly defined planes and directions of slip.

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[1] In certain cases, the type of forces involved are similar: see experiments on bismuth to be described later.

[2] Reference may be made, for example, to the many writings of C. H. Mathewson who has made a special study of twinning phenomena in metals.

Further reference must first be made to the "maximum resolved shear stress" law. Consider a single crystal in the form of a rod of uniform cross-section, $A$, subjected to simple tension due to equal and opposite axial straining forces of intensity, $T$, producing a uniform tensile stress, $f_t$, in the specimen. On any plane, the *normal* to which makes an angle $\theta_p$ with the axis of the specimen, the intensity of normal stress is $f_t \cos^2 \theta_p$. The shear stress resolved along the line of greatest slope contained by the plane has the value $\frac{1}{2} f_t \sin 2\theta_p$, which attains a maximum value when $\theta_p = 45$ deg. when it becomes $\frac{1}{2} f_t$; this is the value commonly used by engineers for design purposes when statistically isotropic conditions are assumed. The deformation of single crystals of the simple type we are considering is *not* controlled by maximum shear stress considerations, because the deformation is restricted to crystallographic planes of a certain type and along certain crystallographic directions contained by those planes. Considering any direction contained by the plane, the inclination of the direction to the axis of the specimen may have any value between 90 deg. and $90 - \theta_p$ deg. Let this inclination be $\theta_d$. Then the value of the shear stress resolved on the plane and in the required direction will be expressed by $f_t \cos \theta_p \cos \theta_d$, its numerical value thus lying between the limits $\frac{1}{2} f_t \sin 2\theta_p$ and zero. Now in the case of a metal which has only one slip plane, this plane may have several contained directions which are crystallographically equivalent and are, therefore, potential slip directions (zinc is an example of this type, having 3 possible slip directions). Again, in other metals, several sets of planes may be crystallographically equivalent, while each such individual set of planes contains several directions which are also crystallographically equivalent. (Aluminum is an example, possessing 4 sets of potential slip planes each of which contains 3 potential slip directions, offering a possibility of 12[1] distinct types of distortion.) In the general case, therefore, we have several potential slip planes inclined at $\theta_p'$, $\theta_p''$, $\theta_p'''$, ..., ..., etc., and several potential slip directions of inclination $\theta_d'$, $\theta_d''$, $\theta_d'''$, ..., etc. A number of resolved shear stress components are thus available, each of the general form $f \cos \theta_p \cos \theta_d$; usually the value of one of these components represents a maximum with respect to the group. If, in such a case, the distortion is controlled only by this *maximum* component, the maximum resolved shear stress law is said to be obeyed. Although slip in metals conforming to the body-centered cubic lattice is not confined to planes of *one* definite crystallographic type, yet a more general form of the maximum resolved shear stress law governs the distortion. In this case, four potential slip directions (the cube diagonals) are available. Let the inclinations to the specimen axis of these slip directions be $\theta_d'$, $\theta_d''$, $\theta_d'''$, $\theta_d''''$. Then, in simple tension, of the single infinity of planes contained by each slip direction, there will exist a plane (not necessarily a crystallographic plane of low Millerian indices) on which the shear stress resolved in the given slip direction will be a

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[1] Even this statement can be challenged in the case of alpha iron.

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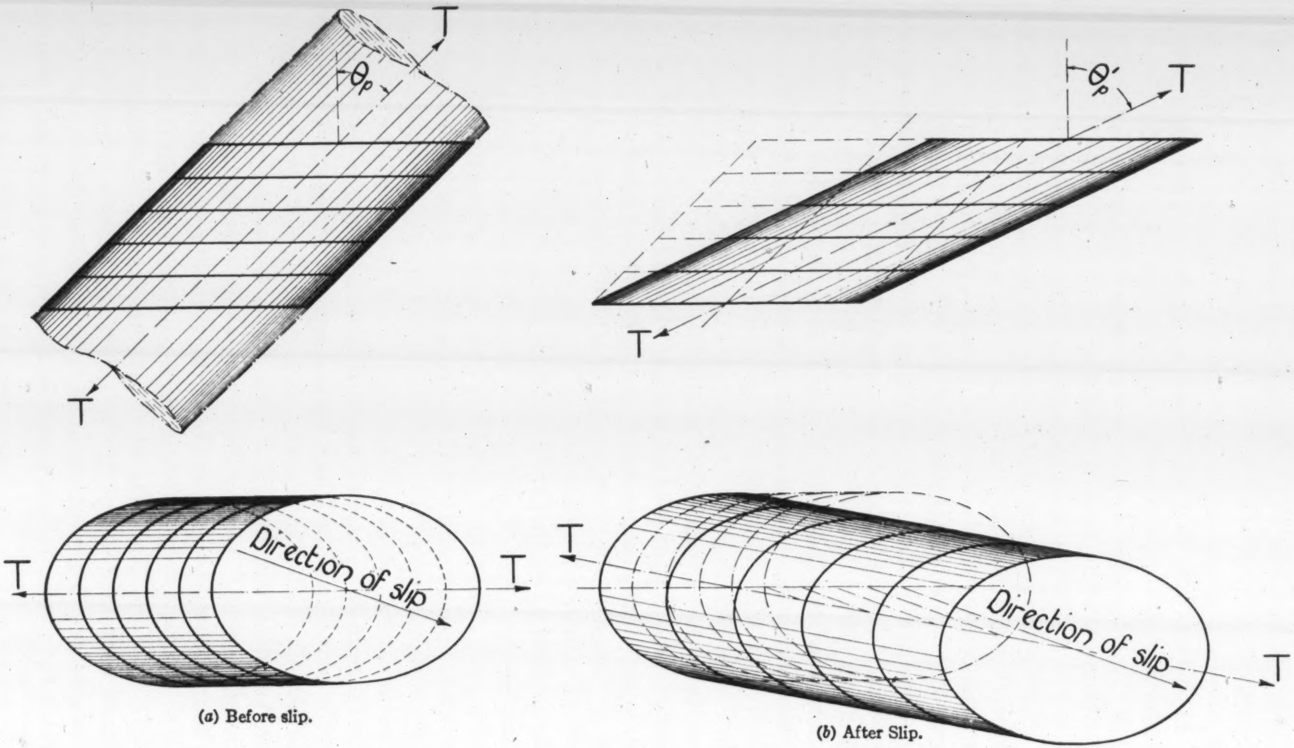
[1] **If attention is paid to the *sign* of shear and of direction, 24 is the actual number involved.**


T
$\theta_p$
T
T
$\theta'_p$
T
Direction of slip
T
T
T
Direction of slip
T

(*a*) Before slip. (*b*) After Slip.

FIG. 2.—Illustrating the Change in Orientation of the Specimen and Crystallographic Axes Caused by Uniform Simple Shear Parallel to a Crystallographic Plane and in a Contained Direction.

maximum. It is apparent that the normal of this plane will make an angle, $(90 - \theta_a)$ deg. with the axis of the specimen, the value of the resolved shear stress being $\frac{1}{2} f_t \sin 2\theta_a$. Thus the four resolved shear-stress components concerned will be: $\frac{1}{2} f_t \sin 2\theta_a'$, $\frac{1}{2} f_t \sin 2\theta_a''$, $\frac{1}{2} f_t \sin 2\theta_a'''$ and $\frac{1}{2} f_t \sin 2\theta_a''''$. Distortion is then found to occur according to the maximum component, and the maximum resolved shear stress law is again obeyed. Thus it can be said that this law is of general application in the plastic deformation by slip of ductile crystals; it has been found to apply equally under tensile and compressive straining forces.[1]

It will be necessary to refer to the change in relative orientation of the specimen and crystallographic axes which results from a process of deformation by simple shear on definite crystallographic planes in contained directions and a brief explanation of this action may, perhaps, be usefully made. The sketches of Fig. 2 will serve to demonstrate the more important aspects. During the process of homogeneous shear on parallel planes, the *area* of each slip plane remains constant. The normal section of the specimen, originally circular, becomes elliptical; the major axis of the ellipse, becoming in the general case, greater than the original diameter of the section. The slip plane tends to move into coincidence with the axis of the specimen, also the slip direction moves towards the line of greatest slope of the slip plane. Considering the edges of, also the normal to, the page on which Fig. 2 is printed, as three rectangular coordinates, it will be seen that the sketches have been drawn so that the slip planes have suffered no rotation with regard to these coordinates, but that the deformation has resulted in rotations of the axis of the specimen in respect to our assumed axes, and hence to the crystallographic axes.

It has become common practice to demonstrate the changes of the relative orientation of the specimen and crystallographic axes, during the distortion of a single crystal by simple shear using the crystallographer's stereographic net.[2] As an example, we may consider a crystal of aluminum. The stereographic projection of the cubic system can be divided into 24 crystallographically similar spherical triangles, each representing the smallest symmetry element of the system; the corners of the triangle denote the positions of normals of planes of the cube, octahedral and dodecahedral types,

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[1] As will be seen later, it also covers the more complicated case of torsional straining, which is, perhaps, the most convincing example of the general application of the law; the force resolution is naturally more complicated, but has been solved, analytically, by Gough, Wright and Hanson(**62**) and has proved a powerful weapon in the study of the fatigue phenomena exhibited by single crystals.

[2] The possibilities of this method, in connection with metal crystals, were first demonstrated by Taylor and Elam(**140**, **141**).

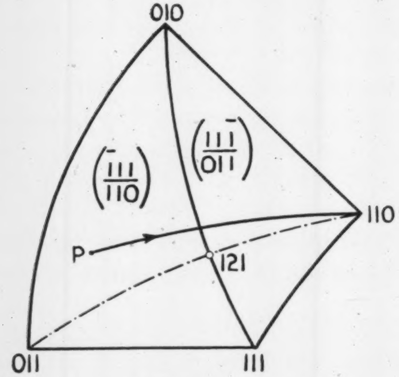
010
(1̄11/110)
(111̄/011)
110
P
121
011
111

FIG. 3.—Stereographic Representation of Distortion.

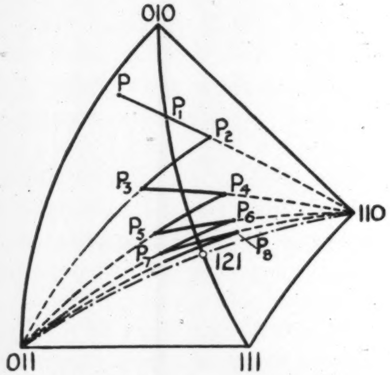
010
P
P₁
P₂
P₃
P₄
P₅
P₆
P₇
P₈
110
121
011
111

FIG. 4.—Distortion as Influenced by Hardening.

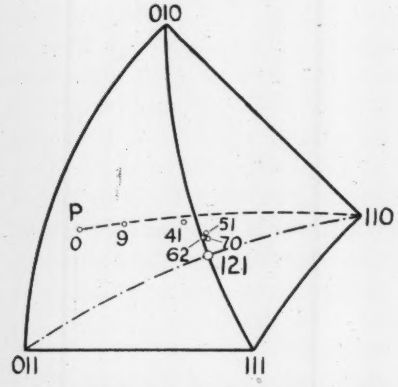

FIG. 5.—Distortion of Aluminum Single Crystal.

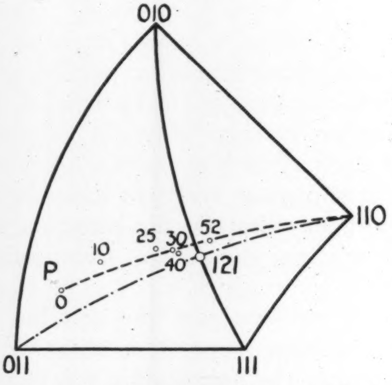

FIG. 6.—Distortion of Copper Single Crystal.

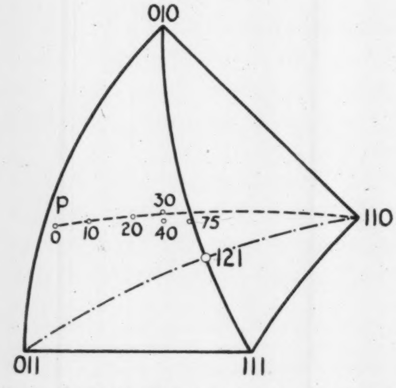

FIG. 7.—Distortion of Silver Single Crystal.

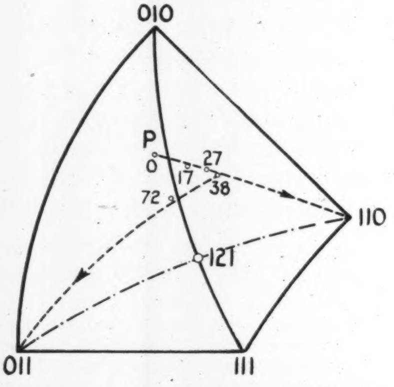

FIG. 8.—Distortion of Alpha-Brass Single Crystal. (70 per cent copper, 30 per cent zinc.)

while the sides of the triangle represent the traces, on the surface of the sphere, of two dodecahedral planes and one cube face. The position of the axis of any specimen can be definitely located with respect to the crystallographic axes using one spherical triangle only: the distortion of a face-centered cubic crystal is, however, more conveniently studied using two adjacent triangles. Figure 3 shows 2 such triangles in which the point, *P*, defines the position of the axis, and, hence, the orientation of the single crystal. If distortion proceeds under tensile straining, then, by the law of maximum resolved shear stress, slip will take place on planes parallel to the $\bar{1}11$ plane and in the 110 direction. As a result of the distortion, the axis of the specimen tends to approach the position of the slip direction and it can be shown, as a geometrical consequence, that the axis *P* will move towards the 110 position along the great circle shown in the diagram.

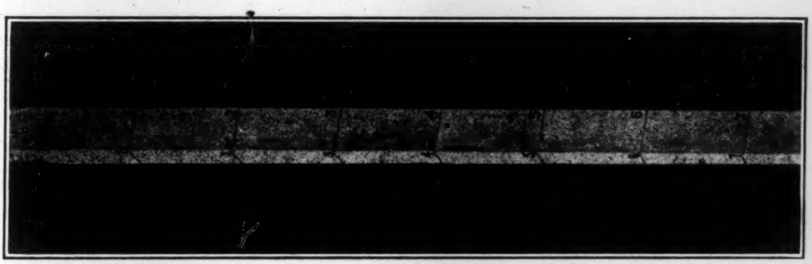

FIG. 9.—Single Crystal of Aluminum After 70 per cent Extension, Showing Uniformity of Distortion.

When a single metallic crystal is distorted by tensile or compressive straining, the uniformity of the resulting change in external shape is very striking. Figure 9 shows the appearance, after an extension of 70 per cent, of an aluminum crystal tested by Taylor and Elam(141); the uniformity of the cross-section is evident and careful measurements of the specimen showed that, up to 20 per cent extension, the variation of similar dimensions in different parts of the specimen did not exceed 2 parts in 1000. But this uniformity of distortion is only an average effect; if we now think in terms of the crystalline structure, it is at once obvious that the actual behavior of single crystals is entirely inconsistent with truly homogeneous shear. For if each crystallographic plane were to take an equal part in the deformation, and if the crystalline structure is not to be destroyed, extensions are only possible which are equivalent to relative movements of neighboring slip planes of an amount equal to an inter-atomic distance (measured along the slip direction) or multiples of that amount. Now shearing movement of these theoretically *minimum* values correspond to enormous distortions,[1] whereas, in actual fact, permanent deformations of *very small* values can be produced at will merely by a suitable choice of straining force. That single crystals do not in fact distort uniformly is shown clearly by the changes in microstructure observed, on the previously highly polished surface of a single crystal specimen, to result from plastic distortion: a typical example is illustrated in Fig. 10. Two characteristic features are always visible in such photomicrographs. The slip bands are very widely spaced (in terms of the unit interplanar distance of the operative slip planes), being separated by areas from which visible signs of distortion are absent; also, individual slip bands are of limited length and do not extend continuously around the specimen. These characteristics suggest that the crystal as a whole is not of uniform strength in parallel directions; also, that the slip planes[2]

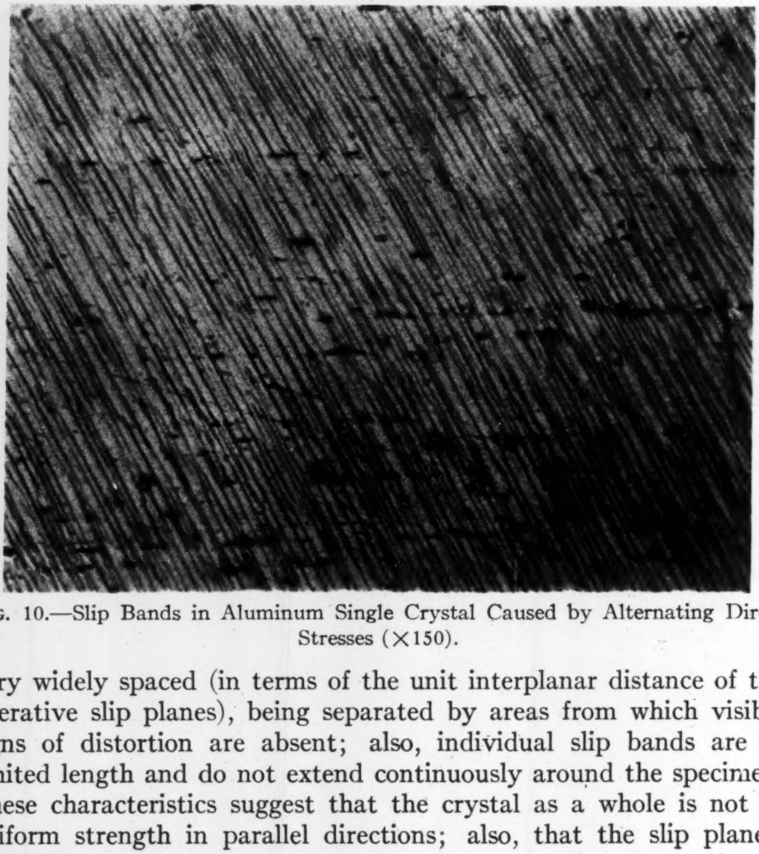

FIG. 10.—Slip Bands in Aluminum Single Crystal Caused by Alternating Direct Stresses (×150).

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[1] It may also be remarked that *uniform* shear movement of this type is entirely inconsistent with the production of *visible* slip bands, for, although the resulting total distortion is large, the resolving power of metallurgical microscopes would not be sufficient to reveal the presence of the slip bands.

[2] It can be stated definitely that the slip bands do, in fact, represent the visible traces of the actual distortion. A very careful and complete series of measurements, made by the author and his colleagues(59), first established the identity of the slip bands with the traces of the crystallographic planes on which the distortion occurred; we have since made many observations on crystals of many kinds with the same result.

themselves are of unequal strength. Another interesting fact is that as the applied straining force increases, fresh slip bands appear in previously undisturbed regions, while the original bands increase in length. Slip bands, at any stage of distortion of a crystal, are not spaced at equal distances apart, but a fairly regular type of distribution is usually observed: measurements made by Yamaguchi(165,169) on aluminum, also by Gough and Cox(73) on silver, indicate that the spacing of the slip bands is, approximately, inversely proportional to the intensity of the resolved shear stress, although the relation may be of a discontinuous type.

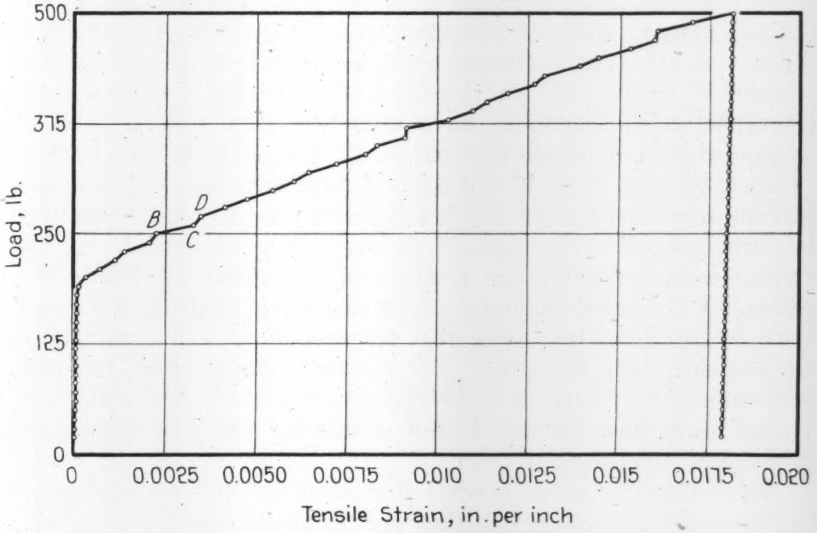

FIG. 11.—Tensile Load-Strain Diagram for Aluminum Single Crystal.

The discontinuous or "jerky" nature of the extension of a typical single crystal is revealed, in a fascinating manner, when observations are made using an accurate optical extensometer; Fig. 11 shows a plot of the data obtained in a typical test(59), made on an aluminum crystal. For example, in this curve the portions *BC* and *CD* corresponded to equal increments of external load, but the resulting strains differed widely. On raising the load to the value denoted by *C*, an appreciable time lag occured before any movement was detected; the specimen then commenced to extend, the rate of extension increasing to a maximum and then decreasing rapidly until movement ceased, the total time involved being about 3 or 4 sec. The movement over the portion *CD* was of a uniform character with no appreciable lag. At neither stage could any further creep be observed if the load were maintained for a long time. The discontinuous nature of slip is affected by the test temperature. Schmid and Valouch(122) record that discontinuous extension of pure zinc crystals is not observed at liquid air temperature. Hanson and Wheeler(84) found that aluminum crystals can suffer considerable plastic distortion at temperatures of 250 and 400 C. provided the deformation is produced sufficiently slowly. They concluded that the mechanism of distortion involved is generally similar (on crystal planes and directions) to that occurring at air temperatures but that the number of slip planes entering into the distortion is much greater while the amount of slip on individual planes is smaller. The available evidence on some of the finer issues, however, is somewhat conflicting and should receive further experimental attention. For example, Yamaguchi and Togino(170) have stretched aluminum crystals at the temperature of liquid oxygen, at air temperatures and at temperatures between 500 and 600 C., and correlated the total extension with the appearance of the slip bands produced. At all temperatures the same general mechanism of distortion was observed. At low temperatures, however, the number of observed slip bands was greater, for the same amount of shear distortion, than that observed at air temperature, indicating that the amount of slip occurring on any one slip plane is smaller. At elevated temperatures, however, recrystallization effects, of a submicroscopic nature, are certain to be of great importance and may lead to conflicting results being recorded by different observers.

Reviewing the above general characteristics of the distortion of ductile crystals, it is clear that they are incompatible with our "ideal" crystal, and several explanations have been advanced to account for these characteristics. Applied to a "static" conception of the atom, the ideas of intrinsic flaws, secondary structure or surface effect may make an appeal. If the problem is considered generally in terms of wave mechanics and quantum theory, the actual planes entering into the distortion and their extent may be determined mainly by "probability" laws, while the spacing of the planes may be determined by considerations of thermal oscillations and frequency of waves of elastic stress. This aspect has been discussed by Finlayson(33). It therefore appears unnecessary for us to assume that fixed flaws or planes of imperfections *must* exist to explain the observed behavior.

We pass on to the useful but mysterious property of the hardening of single metallic crystals following or resulting from static overstraining. Referring to Fig .11 we observe that the resistance of the crystal to extension increases at first very rapidly, then more slowly and is, in general, a function of the extension. On the application of a certain load, the crystal fails by slip and extends; if the load is maintained at some stage this extension ceases and the crystal becomes sufficiently "strengthened" to sustain the load. The assumed properties of our "ideal" crystal offer no prediction or explanation of this phenomenon of strain-hardening. How does it occur? In the first place, if slip on crystal planes and directions is accepted as an observed property of single crystals, then one type of hardening, which may be termed "orientation hardening," naturally results. For we have seen (Fig. 2) that the purely geometrical conditions of homogeneous shear on parallel planes and in a constant direction result in such a relative movement of the crystallographic and specimen axes that the latter would, after an indefinitely great extension of the specimen, tend to lie in the plane and be coincident with the slip direction. If, now, it is assumed that the resistance to slip on the slip plane *remains constant* throughout the distortion, it can be shown from purely geometrical considerations that the required value of the axial stress must increase as the distortion proceeds. It is sufficient to say that the degree of apparent "hardening" due to change of orientation is quite insignificant in amount in comparison with the total amount of actual strain-hardening and may be neglected. In any case, the influence of orientation can be entirely removed

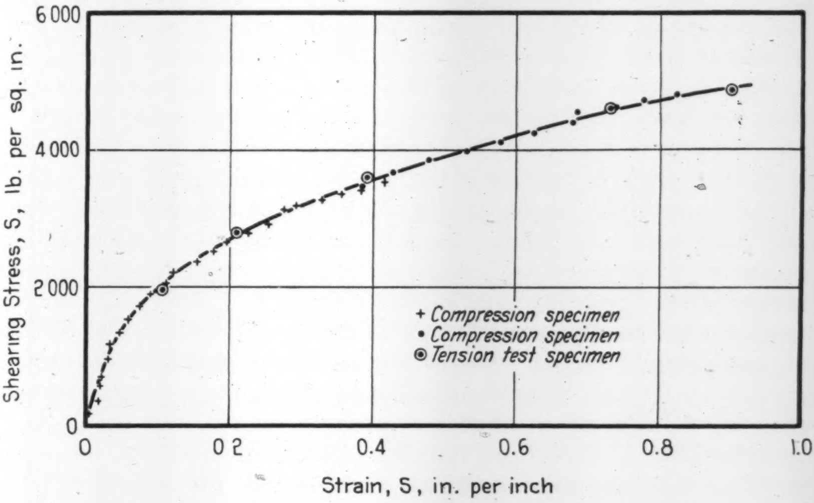

FIG. 12.—Shear Stress-Strain Curve for Aluminum Single Crystal.

from the problem by the use of diagrams in which resolved shear stress is plotted against shear strain, after Taylor(137), also Göler and Sachs(51). Figure 12 shows results of tests made by Taylor which bring out, in a remarkable way, some of the significant characteristics of the strain-hardening of single metallic crystals. A continuous curve fits excellently the results obtained on two crystals of aluminum deformed under compressive loading; also, one deformed by tensile loading. The shape of this curve establishes clearly, in the first place, that marked strain hardening occurs during distortion. The next important point to observe is that, as the results were obtained from different crystals of the same metal, the strain-hardening effects are independent of initial orientation and are associated only with the amount of distortion; strain-hardening must therefore be considered as a real physical property of the crystalline structure of the metal. A significant aspect of this result lies in the demonstration that, whatever may be the causes leading to slip or the particular consequences of slip which lead to strain-hardening, these factors are so characteristic of the metal that they are repeated regularly from crystal to crystal. Again, in Fig. 12 values of resolved shear stress *only* have been considered. Now in the compression tests, the slip planes were subjected to compressive normal stresses, while tensile normal stresses were involved in the tension test. Clearly, the normal stress has had no apparent influence on the slip resistance or mechanism of hardening;[1] similar observation has been recorded for many crystals by Polanyi, Schmid and other investigators; we may conclude that slip is determined almost exclusively by resolved shear stress considerations.[2] Distortion tests have also been made(137) on specimens cut from the same crystal, so arranged that the same slip planes and slip direction entered into the distortion but opposite *senses* of the slip direction; identical shear stress-strain relations were again recorded. There is, therefore, no doubt that the stress-strain relation for a single crystal of a pure metal is definitely related to the crystalline structure of that metal; curves obtained by three independent observers, Taylor(137), Göler and Sachs(49), also Yamaguchi(169), all conform to a relation of the type $s = aS^n$, where $s$ and $S$ denote shear strain and stress, respectively, while $a$ and $n$ are constants.[3]

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[1] Evidence has been advanced to show that the statical *breaking* load of some types of crystal is determined by a critical value of the *normal* stress, but the available data are not considered sufficiently established quantitatively.

[2] As a result of this experimental fact, three-dimensional charts or models have been constructed showing the relation between orientation and ultimate extension; the work of Schmid (**117**) and other German investigators in this field may be referred to.

[3] The experimental values of the coefficient and index vary slightly, but variations in the batches of crystals might account for these: the recorded values of $a$ and $n$ were, respectively, 0.047, 0.013, 0.015, and 2.4, 2.5, 3.0.

Previous examination of slip characteristics revealed that the plastic deformation of crystals is not a process of truly homogeneous shear, neither does each individual slip plane enter into the distortion uniformly over its entire area, yet the resulting mass distortion—as judged by changes of external shape of the specimen—is very uniform; we now observe that shear stress - shear strain characteristics repeat regularly from crystal to crystal. Although all these effects may be, in fact, statistical averages of probability occurrences, they are evidently intimately related to the crystalline structure.

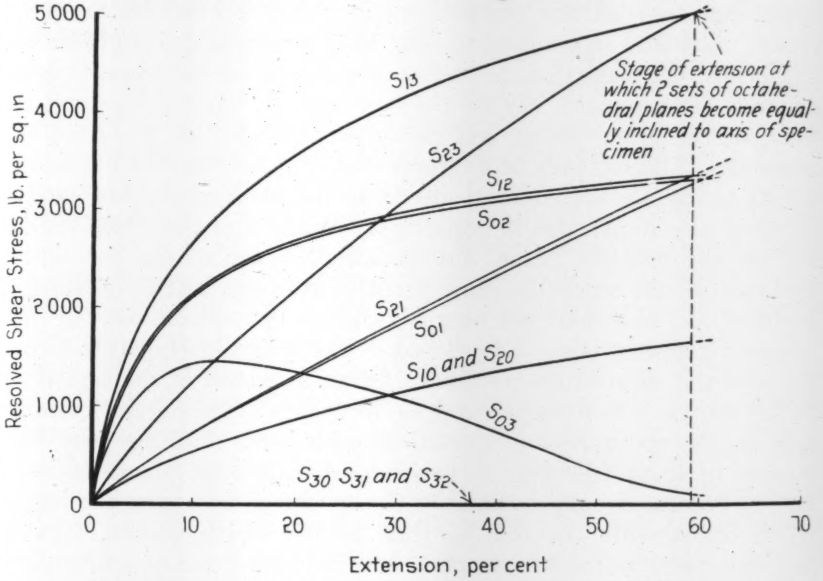

FIG. 13.—Diagram of Resolved Shear Stress on Octahedral Planes During a Static Tension Test on a Single Crystal of Aluminum During that Stage of the Test in Which Slip Is Confined Entirely to One Set of Octahedral Planes.

Plastic deformation occurs by slip parallel to definite planes and directions contained by those planes; it becomes of great interest to examine if strain-hardening is *confined* to the planes of slip; evidence on this point should assist towards obtaining an insight into the mechanism whereby strain-hardening is effected.

Consider a single crystal of the face-centered cubic type, as representing the maximum[1] possible number of alternative but crystallographically similar types of slipping, subjected to axial tensile straining. From the experimental load-extension curve, also X-ray readings, observed changes of shape, etc., occurring during the test, a diagram can be constructed showing the manner in which the values of the stress components vary with the extension. These calculations have been made from the records of a static tension test on a single crystal of aluminum; the results are plotted in Fig. 13. The greatest shear component is $S_{13}$ and, in the test, distortion proceeded by slip on the corresponding plane and direction. Now, we have seen that all the competing slip planes are equivalent with regard to initial resistance to slip and resulting strain-hardening properties, yet distortion, having once commenced[1] on plane 13, was confined to that plane, despite the fact that the stress component on some of the other planes was rising very rapidly as the extension proceeds. This can only be interpreted as evidence that the hardening effects of slip are not confined merely to the plane of slip but extend in other directions; this is important. While it is true that, during slip on the *operative* plane, any individual plane of the competing groups does not retain the same particles as in the initial undistorted position, yet this does not offer a satisfactory explanation why the competing planes become hardened. The diagram (Fig. 13) shows the resolved shear stress conditions for an extension of the crystal of 59 per cent, at which stage, owing to the change in the relative orientation of the specimen and crystallographic axes resulting from the process of shear (see Fig. 2) two sets of octahedral planes (Nos. 1 and 2) become equally inclined to the specimen axis; also the major stress components, $S_{13}$ and $S_{23}$, then assume equal values. Now if hardening was confined to the plane on which distortion had previously taken place, slip on plane No. 2 might be expected to occur *before* the stress components became equal; such "anticipatory" slip has *never* been observed. Again, if the material had become uniformly hardened, the new slip plane should become operative directly equal values of the competing stress components are attained. Also, should hardening be nearly uniform but actually greater in directions not contained by the first operative slip plane, then the change-over of slip plane should be delayed. Thus the distortion of the crystal when studied in relation to the stress analysis provides valuable information regarding the nature of strain-hardening. Test results show that the third alternative is generally obeyed, particularly with alloy crystals, although the second alternative is closely approached in simple metals like aluminum. The actual behavior of typical metals can be examined using the stereographic projection. Referring to Fig. 4 and again using two adjacent symmetry elements then, if the point $P$, representing the axis of the specimen, falls *anywhere* within the left-hand triangle and assuming slip occurs on an octahedral plane and in a contained dodecahedral direction according to the maximum resolved shear stress law, it follows that slip will occur on the $\bar{1}11$ plane and in the 110 direction: $P$ will therefore move towards 110 along a great circle. Similarly if $P$ lies in the right-hand triangle, the slip plane and direction will be $11\bar{1}$ and 011, respectively, and $P$ will move towards 011. In the general case (Fig. 4) as distortion proceeds by slip parallel to one octahedral plane (111) $P$ moves until it reaches $P_1$ on the line joining the points 010 and 111. At any position of $P$ on this line, the axis of the specimen is equally inclined to the two competing slip planes and the slip directions. If the slip resistance of both planes is equal, distortion will now proceed by double slipping and $P$ will move down along the line, 010-111, towards the direction 121; if it reaches this limiting position without fracture intervening, any further distortion will proceed by the same mechanism of double slipping but without involving any further changes in the relative orientation of the specimen and crystallographic axes. Should, however, the operative slip plane at every stage of the distortion be hardened to a lesser degree than the competing octahedral plane, then a process of alternate slipping of the type $P$, $P_1$, $P_2$, $P_3$ ... would result. Typical experimental results are illustrated in Figs. 5, 6, 7 and 8: in each case the number marked on the diagram indicates the percentage extension of the crystal at the stage of the experiment when the specimen axis reached the position[1] shown. Figure 5 relates to an aluminum crystal(141), Fig. 6 to a copper crystal(22) and Fig. 7 to a silver crystal(22). Figure 8, which relates to a single crystal of beta brass (70 per cent copper, 30 per cent zinc), is typical of the behavior of certain solid solution crystals. In general it may be said that, in single crystals of pure metals, slip on one plane persists slightly beyond the position at which the competing plane should become operative if equally hardened, after which slip proceeds alternately on these planes in the manner shown (in an exaggerated manner) in Fig. 4. In alloy single crystals, the tendency for slip to persist on the original slip plane is more marked than in pure metals, as shown in Fig. 8, but it will be seen that the second slip plane also "overshoots" the symmetrical position. Thus Fig. 4 can be regarded as illustrating the general strain hardening behavior of crystals which can be summarized as follows: The process of slipping on a parallel set of planes produces marked increase of resistance to further slip on these planes. Even more remarkable is the fact that the resulting strain-hardening is not confined to the operative slip planes but a *general* hardening of the crystal is produced to such an extent—the actual degree varies in different types of crystals—that the distortion tends to continue on the plane even though a competing slip plane becomes subjected to a greater stress component.[1] A most remarkable property of crystalline matter!

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[1] Three slip directions in each of 4 sets of octahedral planes, or a total of 12 possibilities. Denoting the 4 planes by 0, 1, 2 and 3, and remembering that the principal lines of atoms (or slip directions) represent the intersection of pairs of these planes, we can conveniently represent any slip plane and direction by a combination 01, 12, 23, etc., of which the first figure represents the slip plane, and the second figure the plane whose intersection with the slip plane defines the slip direction. Using this simple convention, the shear stress components will be referred to as $S_{01}$, $S_{12}$, . . , etc.

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[1] At the very commencement of the test, a few slip bands, agreeing with distortion on competing planes, are often observed. This is possibly due in the general case to slight eccentricity of load; it affords additional evidence that the slip resistance of all the slip planes are equal before distortion commences.

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[1] These diagrams do not show the complete movement of the axis owing to the small number of observations made; also, the positions indicated include all errors involved in the X-ray measurements, distortion measurements and eccentricity of loading.

By what mechanism can shear on parallel planes lead to the observed *general hardening* of the crystal? It appears obvious that the consequences of slip cannot be confined to the slip planes: this suggests that comparative examination of the condition of the crystalline structure before and after slip should afford useful evidence in relation to our inquiry. To investigate this aspect, we must turn from mechanical tests and microscopical examination and invoke the aid of X-ray analysis. This aspect will be more conveniently considered at a later stage; in the meantime it is sufficient to state that X-ray analysis does, in my opinion at any rate, supply the answer to our inquiry.

Returning to the stress-strain and geometrical aspect of the statical distortion of single crystals, the discussion has been sufficient to indicate that the property of plasticity in metals is a fundamental issue of the general problem of cohesion; the study of slip phenomena has revealed many characteristics which would be quite inexplicable if encountered in our assumed "ideal" crystal.

Space does not permit discussion of the effect of the addition of alloying elements on slip resistance and strain-hardening characteristics of single crystals; for valuable researches in this important field, the reader is referred to the work of Elam(**21,23**), Göler and Sachs(**50**), Rosbaud and Schmid(**111**), Sachs and Shoji(**114**), Sachs and Weerts(**115**), Karnop and Sachs(**88**), Taylor(**139**) and others.

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[1] A confusion of thought has been evinced by some writers in discussing this characteristic, for stress has been laid on the relative *softness* of the acting slip plane in relation to the competing planes; it has sometimes even been inferred that the act of slipping has made slipping easier. But this is quite contrary to fact, for whatever stage of the distortion is considered, hardening increases with further distortion. The essential fact, with its attendant problem, is indisputable, namely, that planes which have not been previously operative become hardened as the result of a process of shear occurring on planes of entirely different orientation.

## V. THE INFLUENCE OF THE INTERCRYSTALLINE BOUNDARY UPON STATIC STRENGTH AND DISTORTION

One of the aims which inspire the study of the properties of metallic single crystals is, eventually, to correlate these properties with the behavior of the finely-divided crystalline aggregate. In view of the various factors introduced into the problem, it cannot be hoped usefully to effect a *direct* comparison: it appears necessary to make an intermediate study of specimens consisting of two, three and greater numbers of large individual crystals, as, in this way, at least two variables—differing orientation of crystals and influence of the boundary—may be logically treated. At the present time, the study of the properties of specimens consisting of several large crystals represents a field which offers great opportunities for future research; very little work has as yet been performed.

Statements have often been made that an aggregate is always stronger than a single crystal, yet the available evidence on this point is very limited and often unreliable. Before any just comparison can be made, it is essential that the individual grains in the aggregate must be in exactly similar conditions, as regards composition, crystalline structure, internal stress, etc., as the single crystal with which comparison is made; not inconsiderable difficulties are here involved. Again, two other factors may enter into the problem; I refer to the effects of the size of the *specimen*, also, of the size of the *crystal;* these factors may not be related. Wire drawers, for example, are aware of the influence of the first factor in relation to crystalline aggregates but strain-hardening effects are certainly involved. Then the well-known experiments of Griffith(81) are often freely quoted in connection with size effect in metals; in glass fibers of ordinary size (0.04 in. in diameter) Griffith found a tensile strength of 25,000 lb. per sq. in., but as the fiber diameter decreased, so the tensile strength increased; he recorded an experimental strength of 490,000 lb. per sq. in. for fibers having a diameter of 0.00012 in. and, by extrapolation, he estimated that the value for zero diameter should be in the neighborhood of 1,600,000 lb. per sq. in., a close approach to the calculated value of the cohesive strength. To explain these differences of strength in vitreous solids Griffith developed a theory of rupture(82) involving surface tension and crack-formation. But he pointed out the difficulties which impeded the development of the theory to include *crystalline* materials, as, in these cases, the surface tension is not constant, also, anisotropic conditions prevail. I refer specifically to Griffith's attitude towards the interpretation of his own results, because these properties of glass and silica are often applied without discrimination, by others, to problems of crystalline substances. The present position, regarding the effect of size of specimen on strength, appears to be that no certain evidence exists of this effect in metals, whether in the form of a single crystal or a polycrystalline aggregate.

With regard to the effect of *crystal size* on strength, a possible source of misconception may arise from the usual method of estimating "strength," for example, tensile strength, as the maximum load sustained divided by the *original* area of the specimen; this entirely ignores the effect of ductility on the area at which fracture occurs. If a specimen consisting of several large ductile crystals (each occupying the entire cross-section) be stretched to fracture at air temperature, failure, in general, will occur *through* one of the crystals. But the specimen will have distorted to an irregular shape so that an axial section assumes a shape somewhat like that of an hour-glass, the larger dimensions of which represent the unbroken junctions of the crystals. The true stress, at the position of fracture, will be actually greater than in the neighborhood of the boundaries and the experiment has thus demonstrated merely that the boundary restricts plastic deformation rather than revealed any obvious difference in true strength between various parts of the specimen. A ductile single crystal, when strained to fracture, usually draws down to a narrow constriction of very small area at which the value of the true stress at fracture is very high.

Having remarked on some of the possible sources of misconception involved in considering the effect of the intercrystalline boundary on static strength and distortion, the small amount of existing available relevant data may be briefly reviewed.

Carpenter and Elam(15) made tension tests on single crystals of aluminum and on the crystalline aggregate from which these crystals were prepared. They found that the tensile strength of the aggregate was 10,000 to 10,500 lb. per sq. in. with an elongation in 3 in. of 36 to 38 per cent; single crystals gave corresponding figures of 6300 to 10,800 lb. per sq. in. and 34 to 86 per cent elongation. Edwards and Pfeil(26) found that the tensile strength (load divided by original area) of single crystals of iron varied between 21,000 and 34,500 lb. per sq. in., as compared with 44,800 lb. per sq. in. for the aggregate. But they also found that many of the crystals drew down to a "knife-edge" at fracture: obviously, no true indication of comparative strength emerges from these data.

Aston(1) made an investigation into the nature of the distortion in the neighborhood of the intercrystalline boundaries of a specimen of aluminum consisting of 3 large crystals extended by tensile forces.

The extension of each individual crystal produced changes in the relative orientation of the crystallographic axes (in the manner previously described). Aston investigated these changes, near the boundaries, by means of X-ray analyses. He found that the deformation very near to the boundary was extremely small, being much less than the change occurring in the main portions of the crystal; this restriction of slip extended into each crystal for as much as 2 to 3 mm. These valuable observations indicate clearly the influence of boundaries in inhibiting slip; they throw light on observations of Edwards and Pfeil that, unlike an iron aggregate, iron single crystals have neither a clearly defined yield point nor limit of proportionality. Yamaguchi(166) studied the distortion and hardness of aluminum specimens in the neighborhood of the boundaries: the specimens were stretched at different rates of loading and at different temperatures. His results also indicated that the "hardening" effect of the boundaries is due principally to inhibition of slip and this effect was independent of testing temperature or loading rate: no evidence whatever of viscous flow at the boundaries was found.

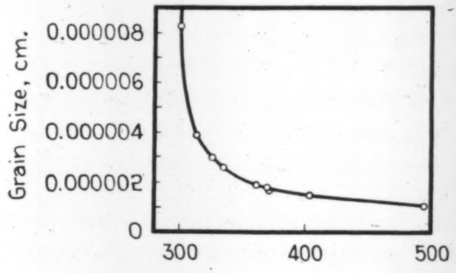

FIG. 14.—Relation Between Grain Size and Hardness.

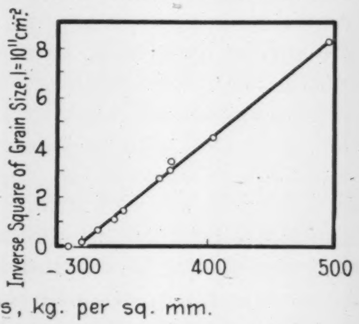

FIG. 15.—Relation Between Grain Size and Hardness.

There exists, therefore, certain indirect evidence that the increased resistance to shear afforded in the neighborhood of an intercrystalline boundary is due to slip interference or inhibition; it should be possible to devise a critical test to examine this idea in relation to varying grain size. My colleagues and I have shown(62) that a pressure indentation into a crystal is produced by exactly the same process of slip on crystal planes as in simple extension or compression. If, then, the presence of boundaries results in an inhibition of slip, comparative indentation tests made on the same material, but in various grain sizes, should show that the hardness is an inverse function of the grain size. Wood(160) has investigated this matter with very definite results. Using a heat-treated steel, he determined the grain size by X-ray analysis. Figure 14 shows the grain size plotted against hardness numbers. It will be seen that, as the size of the crystal grains increases, the hardness decreases in a regular manner. Plotting the results as in Fig. 15, a straight line is obtained, revealing the relation:

$$H = \frac{k}{S^2}$$

where $H$ = hardness number,
$S$ = grain size (diameter of average grain), and
$k$ = constant.

from which it follows *that the hardness number is inversely proportional to the superficial area of the crystal grain.* If to this important result is added the evidence (to be referred to later) afforded by the metallurgical microscope, it appears unnecessary to invoke the assistance of any special hypothesis, such as that of "amorphous" structure, to explain the "strengthening" conferred by such boundaries. This conclusion does not imply that a sudden change of crystalline orientation is effected over an indefinitely thin width of boundary; it may be, as I have suggested(66), that the inter-crystalline zone is occupied by a large number of small crystallites, the orientation of adjacent crystallites being slightly different, and the system being so arranged that the change of orientation from crystal to crystal is achieved by an integration of small changes. Such a boundary composition would be resistant to shear at temperatures far removed from the melting point of the metal but, in view of the lattice distortion which presumably must exist at the junctions of the crystallites, might also be susceptible to stressing at high temperatures, to corrosive influences, and to recrystallization effects. Such a boundary composition appears to be consistent with the interesting experiments of Pfeil(106), who showed that occluded hydrogen had a remarkably weakening (but temporary) effect on the intercrystalline boundaries in iron aggregates, although its effect on the strength of single crystals was negligible.

## VI. EFFECTS OF COLD-WORKING ON SINGLE CRYSTALS AND POLYCRYSTALLINE AGGREGATES

I have previously discussed the general effects of cold-working upon metallic single crystals and it now becomes of considerable interest to examine whether metals, in the form of a polycrystalline aggregate and after subjection to practical forms of cold working, such as drawing, rolling, extrusion, etc., behave in a manner which can be correlated with the deformation of single crystals.

A single crystal of the face-centered cubic type, if extended in tension, tends to approach a limiting position[1] in which the straining axis becomes parallel to a [121] direction. If a bundle of such crystals were placed side by side and then extended simultaneously, the same general crystallographic orientation of the axis would be approached,

TABLE II.—FIBER STRUCTURES PRODUCED DURING COLD WORKING.

| Behavior of Deformed Aggregate | | | | | Deformation Characteristics of Single Crystals, of Same Crystalline Structure, for Comparison Purposes |
|---|---|---|---|---|---|
| | | Observed Fiber Structures | | | |
| Type of Crystalline Structure | Metals Investigated | Axis of Wire or Rolling, Plane of Plate | Normal to Wire Axis or Rolling Direction | References to Publications | |
| | | | WIRE DRAWING | | |
| Face-centered cubic | Aluminum<br>Copper<br>Gold<br>Silver<br>Palladium<br>Nickel<br>Platinum | [111]<br>[111]<br>and<br>[100] | Random | Schmid and Wassermann(**125**)<br>Wood(**157**)<br>Fujiwara(**35**)<br>Greenwood | The axis of a single crystal strained in tension tends to become parallel to a [112] direction. |
| Body-centered cubic | Tungsten<br>Molybdenum<br>Alpha Iron | [110]<br>or<br>nearly | Random | Polanyi(**108**)<br>Fujiwara(**36**) | The axis of a single crystal strained in tension tends to approach a position in which it is contained by a (001) plane and parallel to a [110] direction. |
| Close-packed hexagonal | Zinc<br>Magnesium | (0001)<br>nearly | Random | Not specifically published: mentioned in literature | The axis of a single crystal, strained in tension tends to approach a position in which it is contained by a (0001) plane and parallel to a $[10\bar{1}0]$ direction. The process is complicated by twinning. |
| | | | ROLLING | | |
| Face-centered cubic | Aluminum<br>Brass<br>Copper<br>Gold<br>Copper-Tin<br>Silver<br>Platinum<br>Constantan<br>Nickel | $(1\bar{1}0)$<br>(100)<br>$(1\bar{1}0)$<br>$(11\bar{2})$<br>(100) | [111]<br>[112]<br>[112]<br>[111]<br>[001] | Mark and Weissenburg(**97**)<br>Wever(**151,153**)<br>Owen and Preston<br>Göler and Sachs(**50**)<br>Bachmettew(**2**)<br>Boas and Schmid(**6**)<br>Fukami(**37**)<br>Tamman and Meyer(**135**) | Rolled single crystals of aluminum gave the following fiber structures:<br>*Rolling plane* *Rolling direction*<br>(001) and [110]<br>(110) and [112]<br>(110) and [111] |
| Body-centered cubic | Alpha Iron<br>Molybdenum<br>Tantalum<br>Tungsten | (100)<br>(112)<br>(111) | [110]<br>[110]<br>[112] | Boas and Schmid(**6**)<br>Kurdjumow and Sachs(**89**)<br>Wever(**151,152,153**) | In a single crystal compressed between two parallel plates, the final orientation approaches that in which the normal to the plate is contained by a (100) plane and tends to become parallel to a [110] direction. |
| Close-packed hexagonal | Zinc<br>Magnesium | (0001) within 20°<br>$[10\bar{1}0]$ at 20°/60° | .......... | Schmid and Wassermann(**126,127**) | The axis of a single crystal, strained in tension, tends to a position in which it is contained by a (0001) plane and is parallel to a $[10\bar{1}0]$ direction: if strained under compression, between parallel plates, (0001) planes would tend to become parallel to faces of these plates. The deformation, however, is usually complicated by twinning effects. |

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[1] See section on The Distortion of Single Metallic Crystals Under Simple Static Stressing Systems, p. 20.

although the crystallographic orientation *about* the straining axis would be random depending on the chance initial positions in which the crystals were placed in the bundle. This is the simplest case of "preferred orientation" or "fiber structure." The theoretical fiber structure resulting from the *compression* of a bundle of face-centered cubic crystals is such that the axis tends to lie in a cube face (100) and to approach a position of coincidence with a dodecahedral [110] direction; other simple theoretical fiber structures, based on the geometry of the deformation of single crystals, can be deduced for other crystallographic systems. But there are essential differences existing between our assumed loose bundle of individually free crystals and the grains constituting a polycrystalline aggregate; these render it improbable that their behaviors can be simply and directly related. For example, the deformation of the individual grain in the aggregate must be controlled by the conditions of strain of the specimen as a *whole*, otherwise cohesion would be broken down at the boundaries (and this does not usually happen); also, the deformation may be complicated by such effects as twinning;[1] again, the forces applied in manufacturing processes are usually very complex involving influences such as friction of the dies, rolls, etc.; these must produce non-uniform distortion across the section of the wire or plate. When these facts are taken into consideration, it is not surprising to find that, in many cases, the fiber structure of an aggregate, after cold-working by a complicated process, is not directly comparable with that of a free single crystal after subjection to a uniform distortion by an applied simple straining action. In order to effect a just comparison, we must be able to compare the final structures of single crystals and aggregates after subjection of both to exactly similar operations, say, rolling, wire-drawing, extrusion, etc. Unfortunately, very few truly comparative experiments of this type have yet been made: a notable example is the work of Tanaka (136), who studied the effects of rolling on single crystals of aluminum. He found that definite fiber structures resulted, directly related to the rolling plane and direction, but the final positions were influenced, as would be expected, by the initial orientations of the crystals with respect to the rolls. As shown in Table II, the final structures of these crystals resemble closely those of the crystalline aggregates after similar rolling. Much careful investigation of the final structure of cold-worked aggregates has established conclusively that the attainment of a definite fiber structure, often of a complex nature, is characteristic of such materials. In some cases of rolling, a *duplex* fiber structure results which is equivalent to a mirror image about a rolling plane containing the axis of the specimen. Often, in wire-drawing, rolling, etc., the orientation of the structure at a point depends upon the position of the point in relation to the axis of the specimen; this is merely an effect of the non-uniformity of the distortion imposed by the external straining forces. In view of this complexity, it is impossible to attempt here, in the space available, an adequate summary of the fiber structure of deformed metals. *The essential conclusion in the present connection, however, is that the deformations occurring during these industrial processes of the cold-working of aggregates are intimately related to the crystalline structure of the individual grains; also there is little doubt that the deformation*[1] *of each grain obeys the same general geometrical laws of distortion as those found to control the plastic flow of free single crystals, and, if the straining forces were accurately known, the final structure would be determinable, although much further research is required definitely to establish this correlation.* This consideration forms a further indication that the study of the characteristics of deformation of metallic single crystals is not merely of academic interest but possesses considerable practical importance, both actual and potential.

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[1] Zinc is an excellent example. This material possesses initially only 3 slip movements (1 plane and 3 directions). But by primary twinning, it can produce 6 new sets of potential slip planes and the process is capable of repetition by secondary, tertiary, etc., twinning.

Some typical data,[2] relating to the fiber structure produced in representative metals during the processes of cold-drawing and cold-rolling are presented in Table II; in this connection it may be remarked that the observed final fiber structure is always far from perfect, also, in many cases, it represents the superimposition of two or more structures. But the approach to a state of preferred orientation is clearly a general characteristic of severely cold-worked metals. An important practical aspect of fiber structure is that the material, unless properly annealed, becomes endowed with definite directional properties which are clearly revealed by mechanical and other tests: this is especially pronounced in rolled sheets. The data of Table II also show that, using the same metal and general type of deformation, different investigators have observed different types of fiber structure. But this is not surprising, for it is evident that the final orientation would be sensitive to the applied conditions and extent of the deformation, such as, for example, the severity of the pass in rolling; thus, Taylor and Quinney(146) have shown that the fiber structure of the outer layers of a drawn wire is affected by the number of draws, taper of the dies, etc. Fiber structure is, of course, determined by X-ray methods which are sufficient to demonstrate unmistakably that severe cold-working fails to destroy the essentially crystalline nature of metals; even the thinnest beaten foils(9) give X-ray reflections characteristic of this crystalline structure. These facts offer a striking commentary on the validity of some of the older hypotheses, such as the amorphous theory and that of Czochralski(19), which suggested that the crystalline state can be entirely destroyed by severe cold-working.

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[1] In certain cases, particularly the soft metals, recrystallization may occur during the deformation; this introduces a new factor.

[2] The reader is also referred to a paper(90) by Körber for a summary and discussion of researches, carried out at the Kaiser-Wilhelm-Institut für Eisenforschung, Düsseldorf, into the plastic processes involved in industrial methods of manufacturing materials; for the mechanics of the plastic state, Nadai's comprehensive book(102) on Plasticity should be consulted.

In the present section, attention has been given merely to what may be termed the "geometrical" aspects of cold-working by which the originally random orientation of the crystals has been changed to a preferred orientation of a definite type or types. But a mere *translation* of structure would not be accompanied by the observed marked strain-hardening and condition of severe internal stress[1] which are characteristics of a cold-worked material in its final condition. It is of great interest to examine how these characteristics are acquired. A few years ago, we were restricted to generalizations, mostly of a qualitative nature. Recently, however, the influence of cold-working on the crystalline structure has received intensive study, employing those monochromatic X-ray methods which furnish *quantitative* data; as a result, we are now in a position to revise our earlier ideas and place them on a surer foundation. As much of the X-ray work referred to has been carried out in connection with cold rolling (discussed above in a general manner) it will be convenient at this stage to review this newer knowledge and examine to what extent it can be correlated with observations on slip, also, on the stress and strain characteristics of single crystals and aggregates.

## VII. COLD-WORKING IN RELATION TO CRYSTALLINE STRUCTURE

It is the study of the lines in the X-ray spectra formed by the reflection of *monochromatic* X-rays that has furnished quantitative data regarding the effects of cold-working on the crystalline structure of metals; the work of Wood(154,155,156,159,160,161) at the National Physical Laboratory should be especially mentioned in this connection.

Commencing with a thoroughly annealed polycrystalline aggregate, a careful study of the changes in the X-ray spectra observed at various stages of a cold-working operation reveals the existence of some of the following characteristics: (*a*) *Crystal Break-up:* this term is applied to a reduction in the size of the crystal grains by a process in which the original grains are broken up into smaller crystallites of differing orientation; (*b*) *Lattice Distortion:* the term will be restricted to a deviation from the ideal lattice parameters as measured by the equivalent percentage change in parameter; (*c*) *Preferred Orientation or Fiber Structure:* in which state the crystallographic axes of the crystals tend to assume a constant direction or directions, in place of the original random orientation; and (*d*) *Variation of Electronic Distribution Around the Atoms:*[1] by which the statistical distribution of the electrons around the atom is altered. These four phenomena are independent and are separately identifiable, although the X-ray technique involved is very difficult. Recent research has now shown that the following events are associated with the cold-working of an annealed metal or alloy:

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[1] For example, observe the stress distribution in drawn brass rods as determined by Sachs(**113**).

The first and immediate effect is the alteration in grain size by crystal break-up to an approximately uniform grain size[2] of the order of 0.001 or 0.0001 cm. This process occurs whether the material is initially in the form of a single crystal or a polycrystalline aggregate and is independent of the original grain size. The evidence for this effect is based on the facts that (*a*) the spectrum lines are discontinuous and "spotted" if the grain size exceeds approximately 0.01 cm.; (*b*) if the grain size lies between 0.001 and 0.0001 cm., the lines are smooth, sharp and continuous; (*c*) the lines are broad if the grain size is less than 0.0001 to 0.00001 cm., approximately, broadening "to infinity" as the grain size approaches 0.0000001 cm. It is a most significant fact that, during the cold-working of annealed metals, the lines pass through stage (*a*) to stage (*b*) and, in general, *suffer no further change;* in some metals, it is true, the lines tend to enter into stage (*c*) but *never* to a sufficient extent to show ultra-fine grain size. At air temperatures, therefore, pure metals or alloys appear to be incapable of further crystal break-up beyond a certain limiting stage:[3] *this limit represents an arrangement of crystallites whose individual lengths are of the order of 10,000 inter-atomic distances.*

The second effect is the occurrence of lattice distortion which either commences with the distortion or follows the completion of the first process of crystal break-up: lattice distortion effects are exhibited by line broadening in diffraction photographs taken under conditions especially selected to give high dispersion. Results(156) show that the lattice distortion,[4] appearing very early in the deformation, rapidly attains to a *maximum* value as the deformation proceeds. This maximum value, which is characteristic of the particular metal, has been termed by Wood the "lattice-distortion limit." For many metals and alloys, the value of this limit is of the order of 0.4 per cent change in equivalent parameter, but for aluminum, platinum, molybdenum and zinc, also the self-annealing metals like lead and bismuth, the limit is negligibly small. A typical example of the attainment of a limiting lattice distortion is shown in Fig. 16, which illustrates the behavior(154) of Mumetal (70 per cent nickel, 25 per cent iron, 5 per cent copper); similar curves were obtained from copper, nickel, constantan and transformer steels.

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[1] A particular type of lattice distortion if the latter term is used in a more general sense.

[2] Certain "soft" metals, such as lead and bismuth, form partial exceptions to this rule: owing to recrystallization occurring *during* the process of deformation, a large grain size persists.

[3] It is possible that, at this limiting size, recrystallization forces effect a balance with the forces tending to produce disruption.

[4] Wood has also found that planes of different crystallographic types do not distort equally or at the same rate.

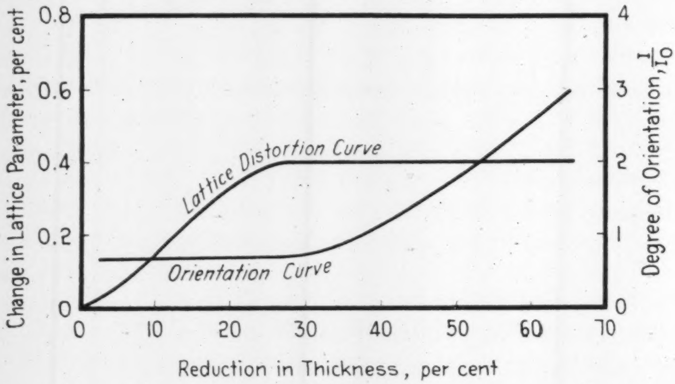
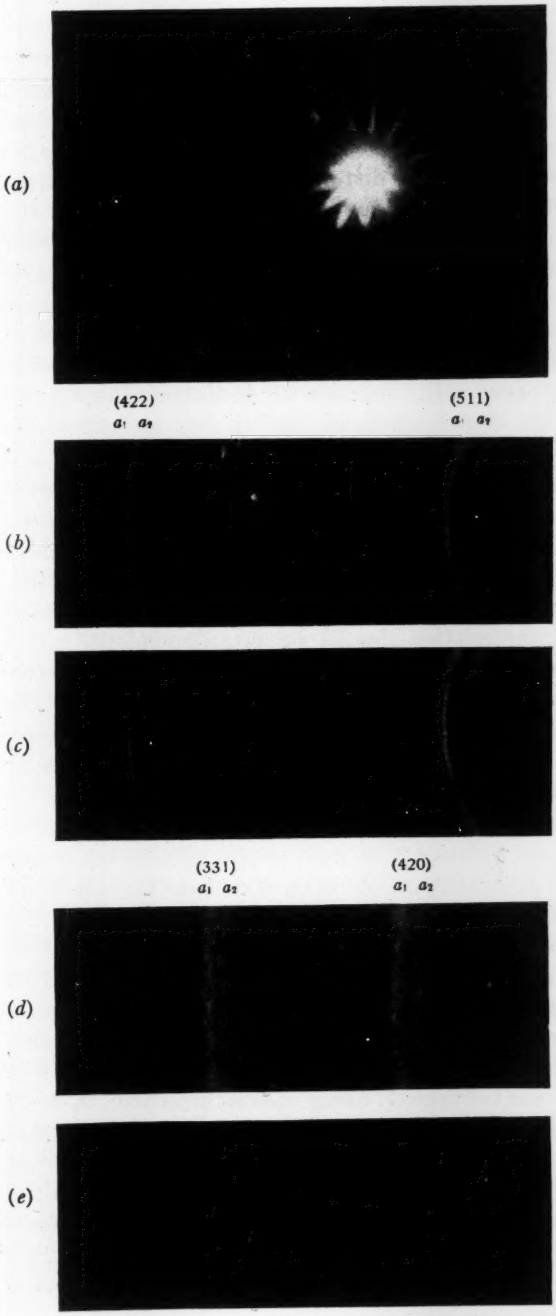


FIG. 16.—Changes in Lattice Parameter and Orientation During Cold-Rolling (Mumetal).

The third effect produced is the appearance of preferred orientation or fiber-structure. As long as the grains in a polycrystalline aggregate are oriented at random, the diffraction lines, obtained in X-ray photographs using monochromatic radiation, are continuous arcs or rings having uniform intensity from point to point. With the onset of orientation, however, the intensity ceases to be uniform along the rings; it is strengthened in some rings at the expense of others. From the angular position of the maxima, the *nature* of the selective orientation can be calculated; from the ratio $I/I_0$ of the intensity of a maximum to that of a neighboring minimum, the *degree* or amount of orientation can be estimated. Thus not only can preferred orientation be measured, but it is clearly distinguishable(154,158) from lattice distortion or crystal break-up. Research shows that preferred orientation only occurs after severe working, such as after 30 to 50 per cent reduction by cold-rolling or drawing; it has



Laué photograph of single crystal ALUMINUM, after deformation under fatigue stressing.

Before deformation

ALUMINUM

After cold rolling

The $a_1$ $a_2$ doublets are equally sharply resolved before and after working.

Before deformation

CONSTANTAN

After cold rolling

The $a_1$ $a_2$ doublets before working the metal are sharply resolved. After working, the doublets diffuse into single broad lines.

FIG. 17.—X-ray Characteristics of Cold-Worked Metals.

not yet been found to any appreciable extent in polycrystalline wires subjected to simple tension or torsion as fracture intervenes. A very important fact, in the present connection is that preferred orientation *follows* lattice distortion; it is a further interesting fact that preferred orientation *commences* at that stage of the deformation which marks the attainment of the limiting lattice distortion; Fig. 16 is an excellent example of this agreement. Now it is common knowledge that the most marked strain-hardening effects are observed in the *earlier* stages of deformation and the typical example of Fig. 16 shows clearly that strain-hardening cannot be ascribed to a state of preferred orientation; it is, indeed, difficult to conceive why the attainment of a fiber structure should produce strain-hardening. The evidence quoted above may, at first sight, appear to indicate that crystal break-up and/or lattice distortion must be the *cause* of strain-hardening and internal stress. But lattice distortion is certainly not *necessarily* associated with strain hardening for aluminum and molybdenum evince no measurable lattice distortion yet exhibit increased hardness with cold-working. Also, considering materials which behave like Mumetal (Fig. 16), why should a state of lattice distortion confer increased resistance to plastic strain? I know no established reasons why these characteristics should be interdependent and I find considerable difficulty in accepting that they are, in fact, directly related in the sense of constituting "cause" and "effect." In the present state of knowledge, the safest attitude to adopt leads to a concentration of attention on the exceptions to the apparently general rule; the behavior of aluminum and similar metals—in acquiring strain-hardening without measurable lattice distortion—is highly significant. These points are of such importance to us, in our endeavor to understand and interpret correctly the finer characteristics of metals, that some typical X-ray photographs, reproduced in Fig. 17, may be of interest. Figure 17 (*a*) shows a Laué diagram of an aluminum single crystal after cold-working by fatigue stresses. This type of diagram certainly shows that the structure has altered but is incapable of differentiating between crystal break-up, lattice distortion, and permanent curvature of the crystallographic planes. The remaining diagrams were obtained using monochromatic X-rays. Figures 17 (*b*) and (*c*) relate to a polycrystalline aggregate of aluminum, while (*d*) and (*e*) were obtained from an aggregate of Mumetal. In the reflections (Fig. 17 (*b*)) obtained from aluminum in the initial annealed condition, the doublets are clearly resolved: after deformation by cold rolling, these doublets are still resolved (Fig. 17 (*c*))—indicating absence of lattice distortion—although marked crystal break-up and strain-hardening has occurred. But in the case of Mumetal, the doublets, clearly resolved in the annealed condition (Fig. 17 (*d*)), *cannot* be resolved after cold-working (Fig. 17 (*e*)): marked lattice distortion is exhibited.

There is one aspect of X-ray spectra which appears to afford a clue of primary importance: I refer to the effect of cold-working as influencing the *electronic distribution around the atoms.* If this distribution is altered, then the scattering of X-rays by the electrons is also affected and, therefore, the relative intensity of different orders of diffraction spectra is changed. Thus, if the electrons around the atomic nucleus are pictured as a "balloon" of electric charge, then the *deformation* of the balloon will affect the relative intensities of the spectra. Such a deformation could occur with or without any displacement of the atomic nucleus, that is, with or without affecting the lattice parameter; it could thus occur *without the accompaniment of measurable lattice distortion as defined above.* The effect has not yet been systematically investigated, but certain cases have already been encountered where as much as 40 per cent change of intensity of reflection has been recorded due to this cause; I consider it very significant that Wood, also Mark and Hengstenberg, have found that aluminum and molybdenum behave in this way. This problem of hardening is so important that it may be profitable to invoke the assistance afforded by evidence of another type. We have seen that aluminum and copper both harden rapidly with cold deformation, both exhibit the same type of hardening curve (true shear stress-strain curve(**22**)), both suffer crystal break-up and both eventually give a fiber structure. But whereas copper develops a very considerable lattice distortion limit, 0.3 per cent, that of aluminum is so small that it cannot be detected by X-ray methods, but the electronic distribution in aluminum is greatly affected. As a cause of hardening, fiber structure can be excluded from consideration as it occurs after lattice distortion is completed. We may also exclude crystal break-up as the cause of hardening; for if a metal is annealed, after cold-working to a stage at which it exhibits marked strain-hardening, crystal break-up and *lattice distortion, both the hardening and the lattice distortion*[1] *effects disappear but the condition of crystal break-up persists.*[2] It is also impossible to consider the difference in shear resistance due to the *new orientation* of the crystallites as accountable for strain-hardening, for, in the case of a face-centered cubic crystal, for example, it is easily shown that however two neighboring fragments are oriented, the values of the maximum resolved shear stress components of each crystallite cannot be in a proportion exceeding that of 2:1. There is another physical change associated both with hardening and lattice distortion which may help us in our problem. It is usually accepted that an unstrained crystalline state represents a condition of minimum potential energy. Hence a deformation resulting in a strain-hardened condition or a state of lattice distortion should correspond to an increase in the internal energy of the system and this is capable of investigation by a comparison of the heat evolved with the work performed by the external forces in producing the deformation. The experimental difficulties involved in experiments of this type are very considerable but reliable data have been obtained by Farren and Taylor(34); their experiments included tests on copper and aluminum. They found that these metals (also steel and a single crystal of aluminum) behaved in exactly the same general manner. In all cases, the heat evolved was found to be less than the heat equivalent of the work performed on the metal,[1] the ratio of the increase of internal energy to the work done having a constant value for different stages of the deformation: the value of this ratio differed with the material, being 8 to 9½ per cent in copper and 7 to 8 per cent in aluminum (13½ per cent for steel and 4½ to 5 per cent for single crystals of aluminum). We may, therefore, reasonably conclude that it is equally possible that this increase in internal energy is associated with the change in electronic distribution in the case of aluminum as with the lattice distortion in the case of copper.

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[1] The simultaneous disappearance of the hardening and lattice distortion can reasonably be advanced as evidence that they are closely associated and I do not desire to exclude this possibility; I insist, however, that the evidence cannot be regarded as conclusive.

[2] See, for example, the work of Goucher(**52, 53**) on tungsten.

Summing up the above evidence we can write down deformation effects in general as determined by X-ray precision measurements in the following sequence: (1) Crystal break-up to a fairly clearly defined limiting size of grain, whose length is of the approximate order of 10,000 inter-atomic distances; (2) disturbance of electronic arrangement; (3) lattice distortion up to a certain maximum which tends to cause further crystal break-up and, possibly, recrystallization; and (4) preferred orientation or fiber structure, remembering that effects (1) and (2) may be simultaneous or (2) may precede (1), also that in certain metals (3) is not present to a measurable degree. Also it is clear that the process of deformation is accompanied by an increase in internal energy. With regard to the exact cause of hardening, we can at present come to no definite conclusion: the foregoing discussion rather favors the view that of the effects (1),

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[1] This increase in internal energy may properly also be interpreted as evidence of phase change, but I am endeavoring to restrict the discussion to phenomena of which definite quantitative data have been obtained; in the absence of such data, phase change is excluded from present consideration.

(2) and (3), all of which are *associated* with hardening, the disturbance of electronic distribution may prove to be the primary cause; but much further research is required before any definite conclusion can be drawn.

The X-ray evidence previously cited showed that crystal break-up was one of the first consequences of deformation; no evidence was afforded of the crystallographic orientation of the deformed structure in relation to that of the original crystals and to the applied stressing system. This information can only be obtained from experiments on single crystals. Some work on these lines was first reported by Goucher(**53**) who examined tungsten crystals after extension to fracture by static tensile forces. Taylor and the author(**144**) have made investigations of a quantitative nature; the metal used was aluminum, subjected to two entirely different types of stressing, the applied strains being insufficient to cause fracture. Two identical specimens were cut from a single crystal of aluminum, the surface of each specimen being polished. One specimen was extended by static tensile forces to an elongation of 23 per cent, the other was extended to the same degree by cycles of reversed direct stresses of high frequency. Each specimen deformed in the usual way from a circular to an elliptical section and slip band measurements, distortion and X-ray readings, all showed that slip had occurred in both cases parallel to the same octahedral plane and dodecahedral direction. Each specimen was sectioned parallel to the operative slip plane and quantitative measurements made of the intensity of the X-ray reflections from the slip plane. These intensities were measured, at different angles of setting, when the specimen was rotated (*a*) about the slip direction as an axis, and (*b*) about an axis also contained by the slip plane but

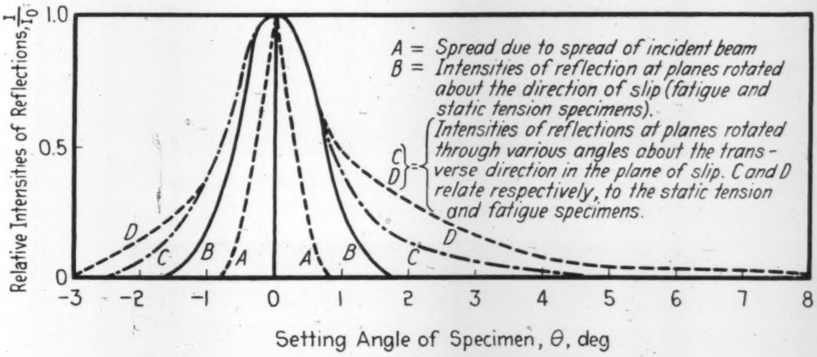

FIG. 18.—X-ray Evidence of Nature of Distortion of Slip Plane.

perpendicular to the slip direction. The results are summarized in Fig. 18, in which $I/I_0$ denotes the relative intensity of the reflection at the stated setting angle to that obtained from the setting giving maximum reflection according to the Bragg relation. The results show that the distortion of the slip plane is very much greater along the direction of slip than in the transverse direction; this general characteristic is exhibited by both the fatigue-stressed and the statically-stressed specimens although the loading conditions were so

FIG. 19.—X-ray Diagram of Tungsten Single Crystal While Strained Elastically at 179,000 lb. per sq. in.

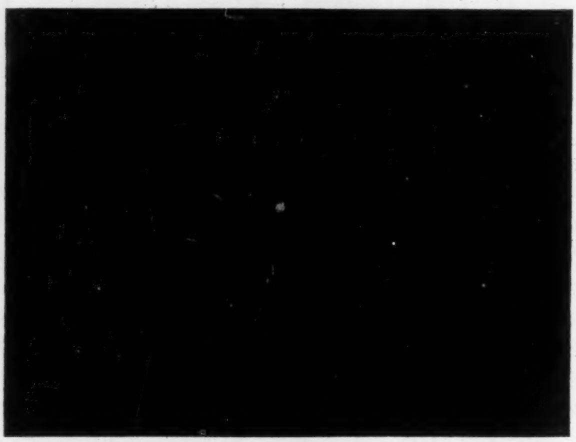

FIG. 20.—X-ray Diagram of Tungsten Single Crystal After Slip Due to Stress of 233,000 lb. per sq. in.

different. ***Expressing this result in terms of crystal break-up, it shows that the crystallites assume orientations which are mainly equivalent to small rotations*[1] *about an axis contained by the slip plane and perpendicular to the slip direction.*** This at once raises the interesting inquiry as to the manner in which neighboring portions of a crystalline body can undergo relative changes in orientation of marked magnitudes without causing fracture. To this inquiry we have no answer that is based on direct evidence; for the rotation of the elements there is experimental evidence but the reason for their rotation remains a problem for the solution of which only hypotheses have been advanced.[2] It appeared reasonable to expect that plastic rotation *might* be preceded by measurable elastic rotation if a sufficient range of elasticity could be provided, and my colleagues, Cox and Backhurst (18) made an experiment to investigate the possibility. If portions of the lattice were affected, the distortion should be evinced by a blurring of the spots obtained by reflections of X-rays from the crystal planes; the crucial test would then consist in determining whether, upon removal of the stress, the X-ray diagram returned to its original appearance before the stress was applied. A tungsten crystal was used as this metal is elastic up to a high stress; Laué photographs were taken at regular intervals at stresses from 4480 lb. per sq. in. up to 233,000 lb. per sq. in. Plastic deformation occurred at a stress of 217,000 lb. per sq. in., clearly shown by large bodily movement of the spots but no definite evidence of lattice rotation was observed in the elastic range. Figure 19 shows the diagram obtained while the material was still loaded elastically, at a stress of 179,000 lb. per sq. in., while Fig. 20, taken at a stress of 233,000 lb. per sq. in. shows the marked permanent change due to slip. The experiment thus yielded a negative result: it showed that, if the elastic rotation occurred, it involved movements which were very small in comparison with the large permanent movements resulting from plastic deformation by slip.

The above descriptions will indicate how enormously our knowledge of the deformation characteristics of metals have been assisted by the use of X-ray methods of precision: the results already obtained suggest that further work on these lines offers a most hopeful method of attack on the remaining problems, although we cannot disregard indications that a stage will be reached when the accuracy demanded of the measurements will exceed the capabilities of the X-ray method.

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[1] In Fig. 18, *positive* values of $\theta$ correspond to directions in which rollers placed between the slip planes would rotate while the slipping is in progress.

[2] I purposely discuss the subject in this pointed manner as I wish to make quite clear the stage of the problem at which precise knowledge ends and at which hypothetical reasoning—however informed and attractive it may be—enters the field in an endeavor to bridge the gap.

We may now turn to the various hypotheses which seek to explain some of the observed deformation characteristics. There is the hypothesis of internal or surface "flaws" or cracks due initially to Griffith(81,82) and developed by Joffé and his collaborators(85). The "mosaic structure"[1] hypothesis of Smekal(128,129,130,131), Zwicky(171,172, 173,174,175), and Goetz(42) is consistent with many experimental observations(4,17). According to this conception, a crystal is pictured as consisting of a repeated arrangement of "blocks," which differ slightly from their neighbors in orientation (each block being inclined at a small angle to its neighbors) or the boundaries between the blocks may be due to a difference in interplanar spacing or to a departure from the average atomic density on parallel planes. The unit block is described as a perfect crystallite surrounded by a very thin "hull" of higher or lower atomic density, giving a "loosened" structure superimposed on the main lattice structure; the boundaries of the blocks are held to constitute the defects responsible for the assumed differences between the theoretical and experimental strengths of crystals. The probable size of the blocks is in doubt but a length of the order of 10,000 interatomic distances has been suggested (it is interesting to note that this figure agrees with the size of the crystallites in deformed metals as determined by X-ray measurements). The "mosaic structure" hypothesis is attractive but must still be regarded as an hypothesis because although several attempts have been made, by Zwicky, to establish theoretical reasons for the existence of such a structure in metals, these have not yet been entirely satisfactory(12,13). Considering the loosened structure hypothesis in the light of our immediate problem, although the nature and size of the proposed blocks may be consistent with the crystallites of cold-worked metals, it is not evident how the *rotation* of these elements is explained. Taylor(138) has given consideration to this problem from the viewpoint of elastic theory. He assumes that the crystal originally contains flaws of finite dimensions and that the material remains sensibly elastic. Then, applying the theory of elasticity, Taylor finds that the following consequences are indicated: Under a shearing stress applied to the plane of the flaw, a stress concentration occurs at the ends of the crack which tends to extend the crack in the same direction but accompanied by an elastic rotation of the material of the same type as is shown by X-rays (for example, see Fig. 18) to occur. Also, as the slipping occurs in a finite number of small local regions, the stress induced at the ends of these cracks may attain a *very* high value even though the applied external forces are moderately small; there is thus reason to conclude that the true strength of the metal is very high and that conceptions of the low strength of metals are based on the misleading basis of the *average* stress which may be sufficient to cause deformation. This process furnishes a plausible explanation of the forces which tend to cause rotation of elements probably leading to crystal break-up. I hesitate to attempt a pictorial representation of the conditions suggested by the Taylor hypothesis but probably they can be visualised somewhat on the lines as indicated diagrammatically in Fig. 21, where *f—f* denotes the presence of flaws and the dotted lines, possibly, the loosened texture of the mosaic structure of Smekal and Zwicky. The sketches indicate the possible condition of the structure before and after slip has occurred. Taylor suggests that his analysis is also sufficient to account, qualitatively, for the form of the hardening curve, and for the constancy of the heat loss ratio during various stages of the deformation. This hypothesis is very interesting and suggestive; detailed comment can only be offered after the full statement is published.[1] One may remark that the two basic assumptions made are extremely debatable, namely, the existence of flaws (whose existence must first be justified) and the appropriateness of the application of the theory of elasticity to areas of molecular dimensions.

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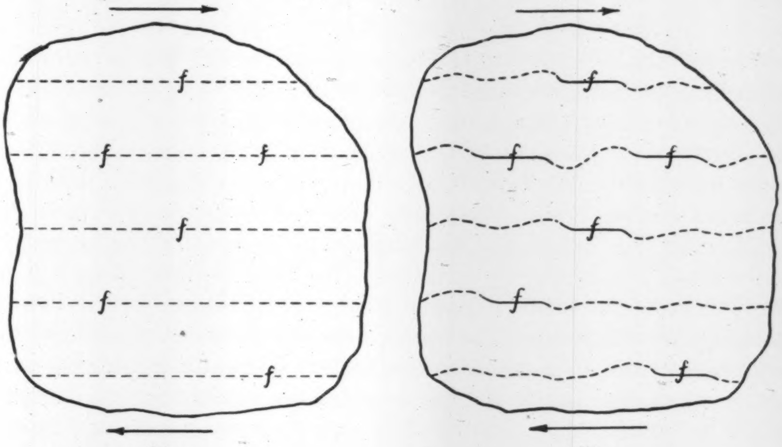
[1] A term originally due, I believe, to Ewald.



(*a*) (*b*)

FIG. 21.—Diagrammatic Representation of Lattice Rotation Resulting from Slip.

Reviewing the facts established by X-ray analysis, we now have a fairly clear idea of the *consequences* of slip on the crystalline structure of metals, but none regarding the conditions under which *fracture* occurs except that the state of the structure at fracture is in a generally similar, though exaggerated, condition to that resulting from plastic deformation. The lattice distortion and degree of crystal break-up attain limiting values at stages remote from the fracture stage, while, although orientation effects may, in some cases, increase right up to fracture, yet, in other cases, fracture intervenes before any appreciable degree of preferred orientation occurs. If we endeavor to obtain information regarding the conditions at fracture from specimens broken under static or impulsive forces, we find that these conditions are of such a complex nature that little useful knowledge emerges.

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[1] The publication quoted (138) was a short summary of the principal conclusions but I understand from Professor Taylor that a complete description is in course of preparation.

In the absence of precise knowledge, I picture the fracture of ductile metals as arising from a consequence of the plastic deformation which precedes the final failure, as follows: Crystal break-up of the original grains must involve strained lattice bonds in regions of the junctions of the crystallites, even though the crystallites themselves be sensibly free from lattice distortion; as the deformation of the grain proceeds, with increasing change in relative orientation of neighboring crystallites, the limiting strain of some of the boundary inter-atomic bonds will be exceeded; this local rupture spreads from bond to bond to form, eventually, the much larger cracks which cause complete fracture. It hardly appears necessary to defend the assumption that, in a disordered structure mentally associated with a state of crystal break-up, regions of severe local strain will exist; also, it is justifiable to assume that such high values of *local* lattice distortion could exist without being detectable by X-ray measurements which record only the *average* value in the grain; further reference will be made, at a later stage, to the suggested relation between crystal break-up and the location of the initial stages of fracture. Further, the argument is not set aside by the fact that polycrystalline metals do not normally fracture at intercrystalline boundaries; for, although the same type of lattice distortion probably exists at intercrystalline boundaries as at the junctions of crystallites, a much greater difference of orientation—and therefore lattice distortion—almost certainly exists in the latter regions. If this hypothesis is correct, fracture will be initiated in those regions which have suffered the most severe plastic deformation, and this is regarded as a critical means of testing the suggestion. For this purpose, we require specimens tested to complete fracture, under conditions in which portions of any one specimen have undergone differing amounts of cold-working. Specially chosen conditions of fatigue testing can be applied to single crystals which exactly meet the required specification and tests of this type (described in the following section) furnish very strong confirmation of the above hypotheses of the effects of slip on crystalline structure in relation to the ultimate failure of ductile metals.

But, in ending this section, I would again sound a note of warning regarding any hypothesis mentioned. We do not understand the nature of atomic structure or why slip occurs; therefore, to explain observed phenomena, hypothesis such as intrinsic flaws, cracks, mosaic structure, etc., are presented. At a later stage, some of the *consequences* of slip appear mysterious: again, certain hypotheses are invoked. All are interesting, some may even prove to be true. *Nevertheless, atomic physics may, in time, prove entirely adequate to account for all the observed phenomena in terms, may be, of electronic distribution or thermal oscillations. As long as we realize that the birth of these hypotheses is due, simply and solely, to our lack of knowledge of the primary problem, we shall not be deluded into a belief that they can be regarded, at the present time, as fundamental, although they may prove to be such eventually.*

## VIII. FAILURE UNDER REPEATED CYCLES OF STRESS, OR "FATIGUE" IN RELATION TO THE CRYSTALLINE STRUCTURE

The study of fatigue in relation to crystalline structure can be divided into two clearly defined periods; it was the availability of large specimens consisting entirely of one crystal which marked the dividing line between these periods and added so enormously to the methods of attack upon the problem of fatigue. Prior to 1923, all that was attempted was a study of the slip band markings and fatigue cracks appearing upon the surfaces of polycrystalline specimens; although the results obtained were of great value, leading to interesting observations, the relation of these slip bands and cracks to the real crystalline structure, being entirely speculative, *could* not afford quantitative data of the type likely to yield fundamental information. The availability of large metallic single crystals, however, made possible the correlation of the applied stressing actions and the visible effects of strain with the crystalline structure itself, as the methods of X-ray analysis could now be employed. In the present section, it is proposed to discuss the results of a series of researches on large metallic crystals, carried out by the author and his colleagues during the period 1923 to 1933; it may be useful to preface this discussion by a few remarks concerning the position at the commencement of the period in question.

Ewing and Rosenhain(29) demonstrated that metals remain essentially crystalline when deformed plastically under static forces, yielding being effected by slip on cleavage or gliding planes within the crystals; also, in some cases, by the production of twin crystals. The first study of the changes in microstructure of metals due to repeated cycles of stress was made by Ewing and Humfrey(28); their observations led them to advance what became known as the "attrition" theory of fatigue. Stanton and Bairstow(133) studied the changes in microstructure of iron and steel subjected to cycles of reversed direct stresses: their observations were in general agreement with those of Ewing and Humfrey and were held to be capable of the same explanation. The "attrition" theory can be summarized as follows: The repeated applications of an unsafe range of stress produces deformation by slip, the nature of a slip band being such that, once formed, repeated slipping can occur between the surfaces forming the slip band. This repeated slipping, being associated with a process similar to that of solid friction, results in the wearing away of the slipped surfaces by attrition, the attrited matter, or débris, being forced to the surface of the specimen, forming heaps on either side of the slipped surfaces. The continued attriting action results in the broadening of the slip band, producing a fissure, which spreads from crystal to crystal as a crack leading to ultimate fracture. It will be noticed that the theory demands that a range of stress which can be applied indefinitely without causing fracture, in other words, one not exceeding the fatigue limit, must necessarily be a range which will not exceed the primitive elastic limit of the metal; also, it introduces the conception of friction between crystalline planes of which it is difficult to find a physical explanation.

Attempts were also made to connect the amorphous hypothesis with the observed changes in microstructure due to fatigue. Beilby(3) suggested that a phase change occurred on the surface of slip which was accompanied by immediate hardening and a state of tension normal to the slip plane: a condition of crystalline layers "sandwiched" between layers of amorphous material being thus produced. If the process was continued under repeated cycles of stress, an increasing amount of the crystalline material was converted, the increasing tensions becoming sufficient eventually to initiate a crack. As a comment on this speculation, it may be remarked that cold-working in general produces a *decrease* in density which might be expected to initiate *compressive* rather than *tensile* stresses across the surfaces of slip. Rosenhain(112) amended somewhat Beilby's conception of the amorphous state by conferring the property of "temporary mobility" on the amorphous material; this leads inevitably to the previous conclusion of Ewing and Humfrey that if slip occurs, even in a few local places, under repeated cycles of stress, then fracture must result if a sufficient number of stress cycles are applied.

But many observations[1] had accumulated which showed that the association of a limiting range of stress with the original elastic state of a metal—as suggested by the attrition and amorphous hypotheses—could not be accepted. Accordingly, in 1920, the study of the effect of fatigue stresses upon the microstructure of metals was reopened in a comprehensive research by Gough and Hanson(57). The changes in microstructure of iron, various steels, copper, etc., occurring under safe and unsafe ranges of direct, bending and torsional stresses were examined in great detail. It was found that marked plastic deformation by slip could occur under a *safe* range of stress, although fatigue failure was always initiated in a region of slip; the propagation of the crack appeared to be due to stress concentration effects set up at the ends of the crack. Hardening occurred under stress ranges less than, equal to, or greater than the fatigue range, indicating that slip was not in itself a weakening process: the initiation of the crack was ascribed to the capacity of the material for strain-hardening having been exceeded locally. A tentative theory of fatigue was proposed but it was recognized freely that many important problems remained, including the following: On what crystallographic planes, if any, does slip occur? What are the stress conditions producing slip? What is the connection between slip and fatigue? What is the influence of the intercrystalline boundary? Are there any common laws governing the fatigue behavior of metals crystallizing in various crystal habits? What conditions lead to the initiation of a fatigue crack? Further study of polycrystalline specimens appeared unlikely to throw much light on these and other problems: we decided, therefore, to commence a study of metals in their simplest form—the single crystal.

In 1923, a commencement was made of the study of the fatigue properties of single crystals of (practically) pure metals. The proposed general scheme of the research was to study in detail the fatigue behavior of typical metals conforming to representative lattice structures. The influence of the intercrystalline boundaries was then to be determined, indirectly, by studies of specimens consisting of two or more crystals. At the present time (June, 1933) the position of the research is as follows: Single crystals conforming to each of the four following lattice groups have been examined in detail: (*a*) face-centered cubic (aluminum and silver); (*b*) body-centered cubic (alpha iron); (*c*) close-packed hexagonal (zinc); (*d*) face-centered rhombohedral (antimony and bismuth). A commencement has also been made in the determination of the influence of the boundary by tests on specimens (of aluminum) consisting of two large crystals. Useful information has been acquired by observing the behavior under corrosion-fatigue conditions of a single crystal of aluminum and of a two-crystal specimen of this metal. In all this work, the same general methods have been employed. The specimen, after preliminary machining, is deeply etched and submitted to X-ray analysis; the surface is then given a good metallurgical polish. After some history of fatigue stressing, the surface is carefully examined using a metallurgical microscope. Very accurate methods(**59,65**) have been developed whereby the exact orientation of any visible features (slip-bands, twins, fatigue cracks, etc.)[1] can be measured and photographed with reference to the crystalline and straining axes and thus correlated to the calculated stress analysis. Thus mechanical, microscopical and X-ray methods are all employed, furnishing a powerful combination in the investigation of fatigue in relation to the crystalline structure itself. In attempting a brief summary of the work, historical order will be disregarded.

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[1] For complete discussion see the author's book(58) on fatigue.

*A. Face-Centered Cubic Lattice (Aluminum and Silver):*

A preliminary investigation(**59**) of the general characteristics of aluminum crystals under reversed direct stresses, reversed torsional stresses, tensile impact and ball indentation, showed that the deformation was determined entirely by resolved shear stress considerations. The slip plane and slip direction were of the general types (111) and [110], the deformation of the crystal at any point being determined by the maximum resolved shear stress component at that point. It was established, for the first time, that slip bands represent the traces of the slip planes on the surface of the specimen. The crystals possessed no primitive[2] elastic limits, while hysteresis effects exhibited during slow cycles of repeated tensile loading were in all respects similar to those obtained in tests on crystalline aggregates; thus, strain hysteresis could no longer be primarily associated with the presence of intercrystalline boundaries. When the conditions of the fatigue tests made using direct stresses were such as to cause very considerable extension of the specimen, then the change of relative orientation of the crystallographic and specimen axes, also the change in shape of the specimen as a whole, were exactly similar to those resulting from static tensile forces. During such a distortion, stress analysis showed that the shear stress component *increased*—under the constant external load—although the distortion ceased if the applied range of stress did not exceed a limiting value. It was thus shown quantitatively that fatigue stressing, like simple static straining, is essentially a cold working operation producing strain-hardening of the material; a diagram exhibiting the general features of Fig. 4 was obtained under fatigue conditions, showing that the strain-hardening effects[1] were not confined to the slip plane operative at the moment. Laué photographs revealed that permanent distortion of the crystalline structure was caused by plastic deformation under fatigue, but X-ray analysis using monochromatic rays failed to disclose any lattice distortion; the observed "asterism" was, therefore, unlikely to be due to a constant curvature of crystallographic planes but rather to crystal break-up. (See the section on Cold-Working in Relation to Crystalline Structure, page 46.)

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[1] The observations involved in this work are very considerable. A usual form of specimen has a test portion 0.4 in. in diameter, and up to 1 in. in length. At each stage of the experiment, the whole of this surface has to be surveyed at least once using a metallurgical microscope of high power

[2] "Primitive" denotes that exhibited on the first loading; the term "natural" elastic limits is reserved to distinguish a range of acquired elasticity resulting from a single or repeated applications of a stress cycle. The primitive elastic limit of a single crystal of aluminum was less than 160 lb. per sq. in.

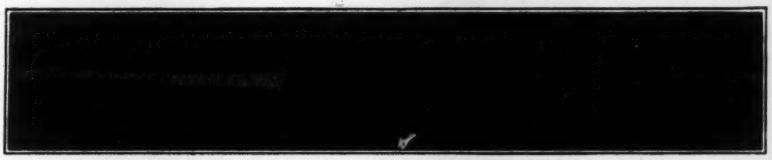

FIG. 22.—Aluminum Single Crystal After Fracture Under Single-Blow Tensile Impact of Very Short Duration.

An interesting experiment(60) will serve to illustrate two important points: (*a*) that large deformations can occur under cycles of a safe range of stress; and (*b*) that strain-hardening is not effected merely by the *first* application of the maximum stress of a cycle but is dependent upon the applied *range* of stress, the final hardened condition thus resulting from a process of *repeated* cold-working. A crystal loaded in tension deformed considerably with marked production of slip bands. Further extension with the production of more slip bands took place during the next 1000 applications of load after which no more movement was detected; a total of 27,000,000 repetitions of load were applied after which the test was discontinued. Under a safe range of stress, the material thus took a considerable number of cycles of loading before strain-hardening was effectually completed; the mechanisms of slip and change of shape of the specimen were found to be exactly similar to that occurring under statical straining forces. Thus, the main phenomena of cold-working under fatigue and static forces are very similar. Now the most marked characteristic of the fatigue failures usually encountered in engineering machine and structural components is the almost complete absence of appreciable distortion of the metal *as a whole* in the neighborhood of the fracture[1] and it was considered desirable to search for a form of fatigue test which would fracture even an extremely ductile single crystal without appreciable change of its external shape. A number of single crystals, of widely differing orientation, were tested in a fatigue machine applying cycles of reversed direct stresses. It was found that nearly all the crystals extended and changed their external shapes under safe or unsafe ranges of stress, the extent of the deformation being dependent on the original orientation: as such a deformation involves considerable variations in the values of the shear stress components during the test, and as this variation cannot be controlled, it was concluded that fatigue tests of this type were not the most suitable for the desired study of fatigue characteristics; another disadvantage is that the extension of the specimen tends to introduce unknown stresses due to eccentricity of loading. We found that a fatigue test in which cycles of reversed torsional couples are applied possessed very great advantages and the majority of the tests made have been of this type. At first sight it is surprising that the apparently complex state of stress conditions arising from the application of torsional couples to a single crystal should afford a useful method of investigation, but actually the distortion and fatigue characteristics followed closely a comparatively simple stress analysis; this became explicable, however, when an analytical investigation**(162)** showed that the stress distribution in a cubic crystal, or in any circular or elliptical cylinder of homogeneous aelotropic material, subjected to terminal couples, is precisely the same as that which would exist in a truly isotropic material. A typical example will serve to show both the special usefulness of the torsion test and to explain many of the general characteristics of fatigue of single crystals. The stress analysis of single crystal torsion specimens was worked out**(62)** with the following

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[1] Although a wide range of testing speeds and frequencies has been employed, no evidence has been obtained of any marked "speed effect" upon the distortion and strain-hardening. In this connection Fig. 22 may be of interest; it shows a single crystal, initially of circular section, fractured under a single blow tensile impact of extremely short duration. The whole process of slip on one plane, followed by alternate slipping on two planes, and necking to fracture took place in this specimen as in a typical slow tension test to destruction.

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[1] This is due merely to the fact that bending stresses are principally involved: single crystals cannot, in general, be tested in fatigue testing machines of the rotating bar type owing to the degrees of freedom for slip not being distributed symmetrically around the axis of the specimen as is the case in a finely divided crystalline aggregate which behaves as if statistically isotropic.

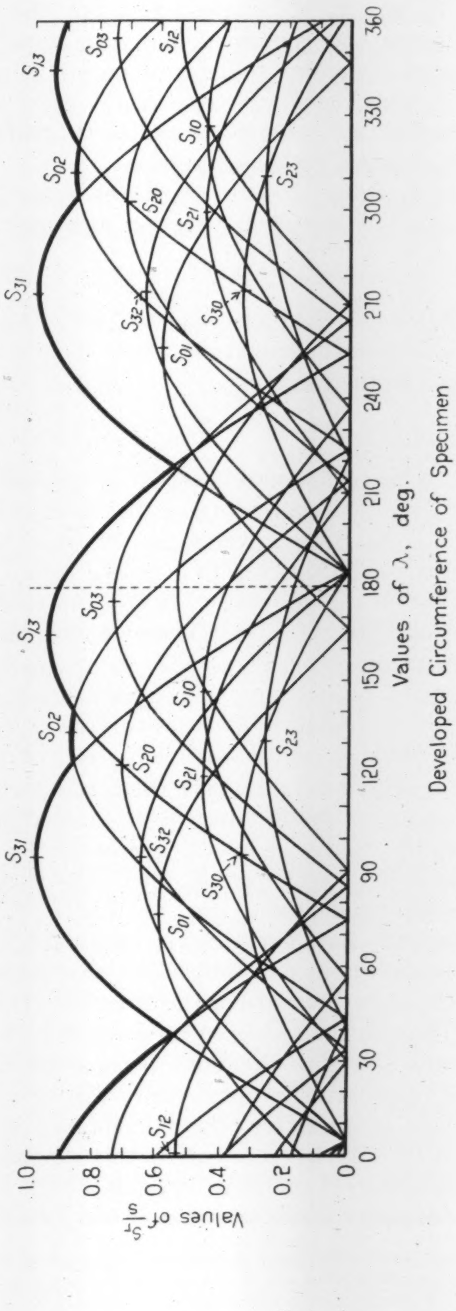

FIG. 23.—Resolved Shear Stress Analysis of a Face-Centered Cubic Crystal Subjected to Torsional Couples.

main result: If $\theta_p$, $\psi_p$, $\theta_d$, $\psi_d$, respectively, are the spherical coordinates of any plane and contained line with respect to the axis and one generator of a circular cylinder of radius, $r$, subjected to a torsional couple, $T$, then the shear stress at any point on the surface of the cylinder, *resolved along the plane and in the contained direction* and denoted by $S_r$, is given by the expression:

$$S_r = [\sin\theta_p \cos\theta_d \sin(\psi_p - \lambda) + \sin\theta_d \cos\theta_p \sin(\psi d - \lambda)]S$$

where $S$ = maximum *nominal* shear stress on surface

$$= \frac{2T}{\pi r^3}$$

and $\lambda$ = angle between axial planes containing the point in question and the reference generator.

This can be reduced to the form

$$\frac{S_r}{S} = A \cos(\lambda - \alpha)$$

where $A$ and $\alpha$ are constants involving only the spherical coordinates of the plane and direction.

Thus the value of a shear stress component will vary from point to point around the surface, according to a simple cosine law, rising to two maximum values in the full circle. A face-centered cubic crystal has twelve slip possibilities, four octahedral planes each containing three possible slip directions. Figure 23[1] shows the stress analysis for a typical specimen, plotted on a base representing the developed circumference of the specimen: under reversed torsional couples, the amplitudes of the various curves will vary from a positive value through zero to a negative value, but their *relative* values remain constant through a stress cycle. It will be noticed that the stress conditions are exactly similar at opposite ends of any diameter and this affords a valuable check on whether any observed surface markings, such as slip bands, twins, etc., are due to the applied stress effects. If the fatigue characteristics are controlled entirely by resolved shear stress conditions it would follow that these would be in accordance with the upper *envelope* of the stress curves; this is found to be the case. Thus, in the specimen to which Fig. 23 relates, three different slip planes and slip directions become operative, and, as the stress on each operative slip plane varies regularly over the area in which that plane is operative, the influence of stress variation can be studied closely. We have, in fact, in this specimen, the equivalent of a simultaneous test on three crystals: such considerations, together with the beautiful demonstration of the general applicability of the resolved shear stress law, enable a torsional fatigue test on a crystal to afford particularly instructive information.

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[1] No attention has been given to the "sign" of the stresses in Fig. 23; these can be determined but are not of importance in the usual case.

The typical fatigue behavior of a single crystal may be briefly described in relation to Fig. 23, first, under repeated applications of a *safe* range of stress. If the applied range is sufficient to produce slip over the whole surface of the specimen, three series of slip bands appear, corresponding to the traces of the three operative octahedral planes, in accordance with the stress distribution shown in the diagram. Thus Fig. 24 shows a typical field at the "crest" of a stress curve ($\lambda$ = 270 deg.) where one slip plane and direction only are operative; Fig. 25 shows a typical "change over point"[1] of the operative slip

FIG. 24.—Slip Bands in Aluminum Single Crystal, Under Torsional Fatigue, in an Area Where One Slip Plane Is Operative (×150).

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[1] The fact that the operative slip plane changes so readily in accordance with the shear stress distribution affords an interesting side light on the true "local" nature of slip. Obviously slip is not a simple process of shear over the *whole* of any set of parallel crystallographic planes; if this were to happen, for example, in the case shown in Fig. 23 where *three* distinct slip planes are operative, we should find visible traces of all three planes at each portion of the surface, and this is not experienced. Thus, the behavior of a single crystal under alternating torsion is probably the most powerful demonstration that slip is such a very local event that it can occur on different planes in different parts of the same crystal without the cohesion of the specimen as a whole being destroyed.

plane, in the region denoted by $\lambda = 300$ deg. With further reversals of stress, the rate of production of slip bands decreases to zero. Thus, the material has become gradually strain-hardened under cycles of a constant range of stress. This process can be examined in detail if the surface of the specimen is polished at intervals during the experiment, the last areas in which slip bands are produced being those corresponding to the peaks of the stress curves. After this strain-hardening process has been completely effected, the stress cycles can be applied for an indefinitely great number of times without any slip bands or cracks being produced. Single crystals undoubtedly possess a fatigue range expressed in terms of resolved shear stress; our experience of aluminum, based on tests extending up to 100,000,000 stress reversals, indicates that this limiting value lies between[1] ±1700 to ±2200 lb. per sq. in. Under an *unsafe* range of stress, the strain-hardening characteristics are generally similar. The rate of production

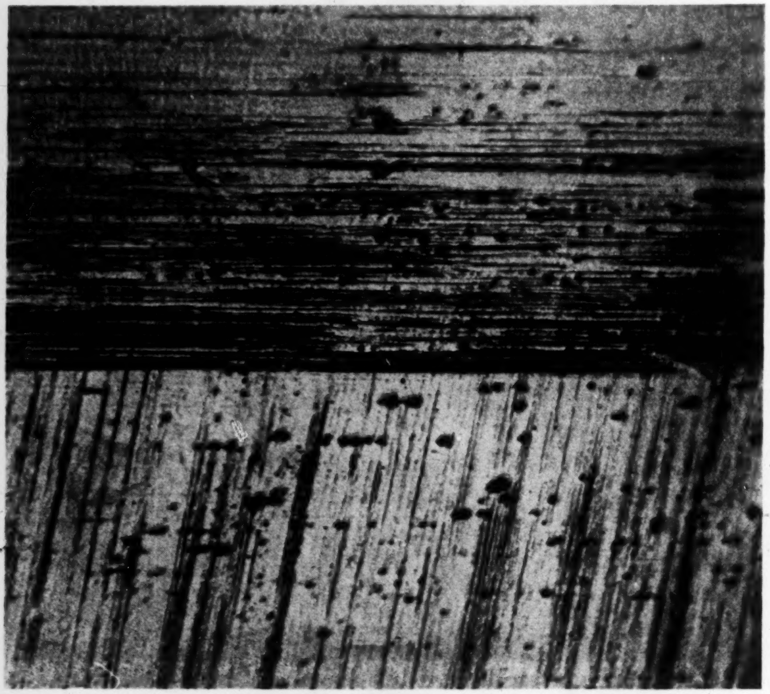

FIG. 25.—Slip Bands in Aluminum Single Crystal, Under Torsional Fatigue, in an Area Where a Change Occurs in the Operative Slip Plane (×130).

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[1] A series of endurance tests are now in hand to establish this value more closely.

of slip bands also decreases with an increasing number of stress reversals and it is certain that the material is strain-hardening irrespective of whether the applied range is safe or unsafe. Generally, however, slip bands are still being produced when visible cracks appear and these cracks are always associated with the regions of maximum resolved shear stress and areas of previous heaviest slip: this fact has emerged clearly from a number of tests on many specimens of ductile crystals of various habits. But it is not *essential* that slipping shall be in active operation when the crack first becomes visible; the specimen to which Fig. 23 refers is a most interesting example of this very important observation. The crystal was tested throughout at a constant range of stress (±2200 lb. per in.) which just exceeded the estimated safe range: complete fracture occurred after a total of 9,270,000 stress reversals. The specimen was examined and repolished six times during the experiment. Progressive strain-hardening was in progress for about 7,000,000 reversals. After 7,200,000 reversals, the production of slip bands ceased entirely, also, a careful examination failed to disclose any visible signs of fatigue cracks. After complete fracture, *no slip bands were visible* and it was thus established that active slip is not essential in the stages immediately associated with the initiation and propagation of the crack. Yet fatigue cracks are *always* initiated in the regions previously subjected to the most severe cold-working. *It was con-*

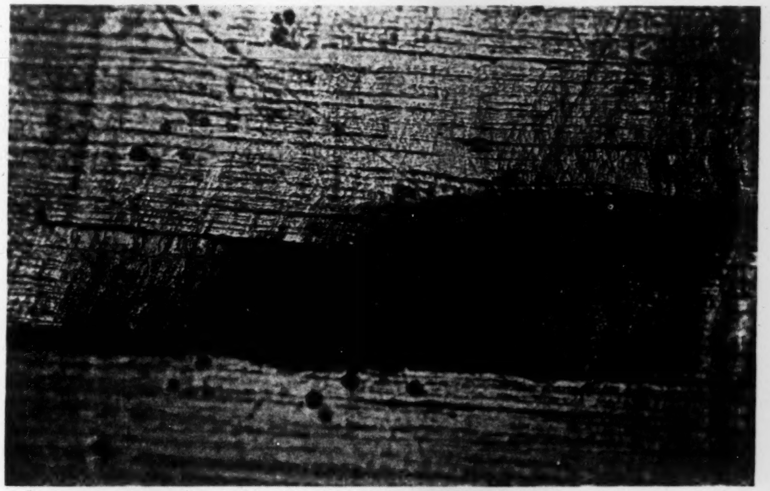

FIG. 26.—Single Crystal of Aluminum After Fracture by Fatigue (×300). Reduced ⅓ in reproduction.

*cluded that slip is the process by which strain-hardening is effected; that, while the limiting fatigue range of stress does not mark the limit of general hardening, yet the amount of local hardening is definitely limited; that the initiation of the fatigue crack is a consequence of the limit being exceeded; that the actual mechanism of cracking consists of the propagation of discontinuities in the crystalline structure arising from the previous strain-hardening.* With regard to the course followed by the crack, the latter, in its very early stages, nearly always coincides with the trace of the operative slip plane on the surface. Figure 26 is an excellent example of this. More commonly, however, the direction of spread of the fatigue crack does not follow any definite crystallographic direction; under alternating torsion, the most common general path is that of the maximum *nominal* shear stress (along or perpendicular to the axis of torsion) while under alternating direct stress, fracture occurs across a section normal to the straining axis. *Thus, while there is no doubt whatever that cracking is initiated along the operative slip plane, the structure appears to be so generally damaged by slip that the subsequent fatigue crack can follow the path of least*

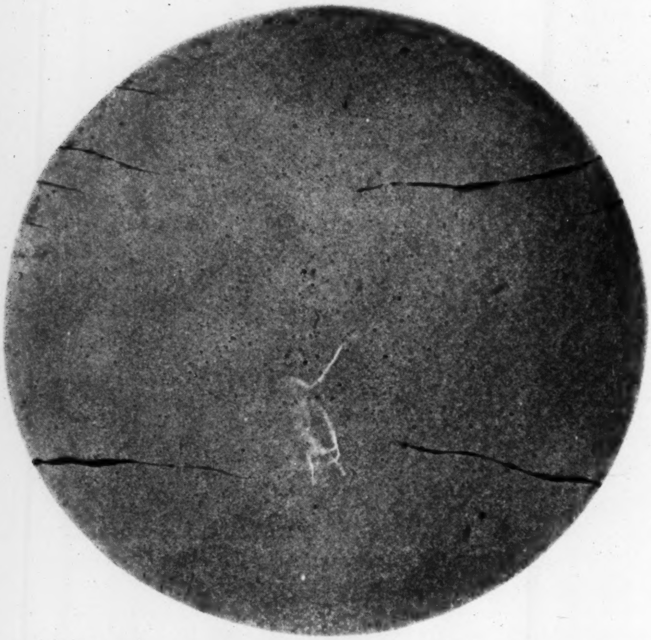

FIG. 27.—Fatigue Cracking in Areas of Greatest Resolved Shear Stress (×10).

*resistance to the external applied forces.* Figure 27 shows the cross-section of a crystal after fatigue failure by alternating torsion: the fatigue cracks are concentrated about the ends of two diameters which correspond to the regions subjected to the maximum resolved shear stress. It was also shown(62), by ball indentation tests, that the effect of alternating stresses on a single crystal is to produce increases in hardness which are roughly proportional to the applied range of resolved shear stress; this applies equally to safe and unsafe ranges of stress.

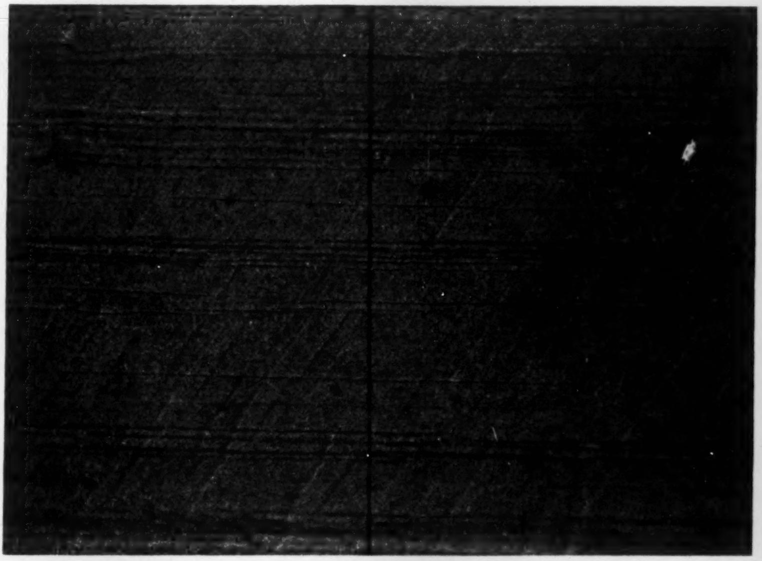

FIG. 28.—Slip and Fatigue in Aluminum Single Crystal Subjected to Combined Stresses (×330).

Under cycles of *reversed* torsional stresses, the maximum range of stress and the maximum numerical value of the shear stress component are inseparably associated. To obtain some information regarding the separate influence of these factors, also of normal stress, on slip and fatigue, two identical crystals of aluminum were tested under combined stresses, alternating torsional stresses with a superimposed static tensile or compressive end load. The results showed that the distortion in the very early stages of the test was determined only by the maximum numerical value of the shear stress component; slip occurred on certain planes. The principal slip however was controlled by the maximum range of resolved shear stress and this maximum range was entirely responsible for the initiation of fatigue cracks. Figure 28 illustrates this dual action in a striking manner, showing slip on two different sets of planes due to the maximum component and the maximum range. The more important fatigue action is clearly distinguishable by the shearing of the material *across* the planes on which the preliminary slip occurred. The normal stress had no appreciable influence on the deformation or on the conditions leading to the initiation of fatigue cracks, but the *propagation* of the fatigue cracks was greatly accelerated by tensile normal stress and retarded by compressive normal stress, resulting in widely divergent endurances of the specimens after the stage at which fatigue cracking was first rendered visible.

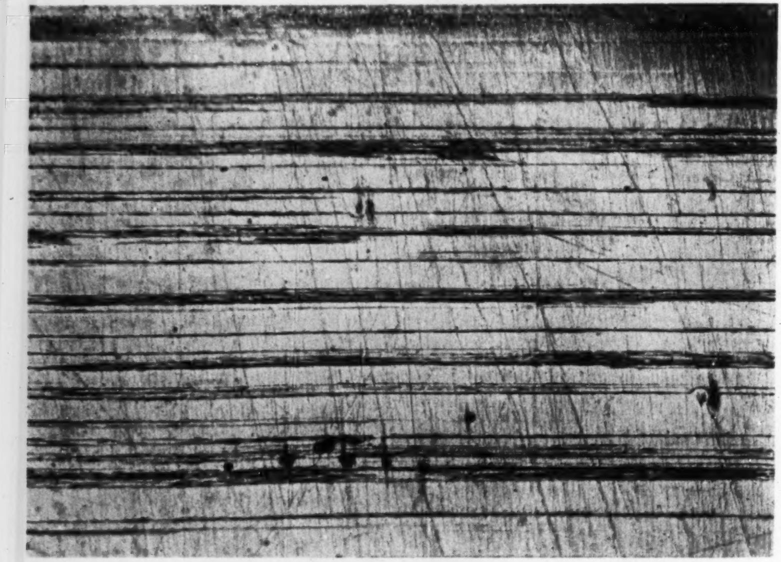

FIG. 29.—Slip Bands in Silver Single Crystal Due to Cycles of an Unsafe Range of Stress (×100).

Fatigue tests have also been made on single crystals of silver (73) and copper. The general fatigue characteristics exhibited by these metals are exactly similar to those of aluminum, the slip band distribution and the initiation of fatigue cracking being controlled essentially by resolved shear stress considerations. Figure 29 shows typical slip bands in silver due to cycles of an unsafe range of stress: similar bands of "massed" slip bands have been found to be associated, in metals of the face-centered cubic lattice, with deformation under stress ranges exceeding the fatigue limit. The behavior of silver crystals received particularly close attention as polycrystalline aggregates of this metal often reveal profuse twinning; it was possible that such a complex stress system as torsion might enable a study to be made of the twinning habits of the face-centered cubic lattice. No twins whatever were formed under torsional fatigue. Attempts to produce twins in silver crystals by compression, also compression followed by impact, were equally unsuccessful, although etching methods, including annealing, were carefully utilized. In every case, deformation took place by the usual process of slip on octahedral planes in the dodecahedral direction. An interesting discussion(73) on these results revealed a considerable doubt whether purely mechanical twins have ever been produced in face-centered cubic metals; if, on the other hand, this is not the case, our experiments indicate clearly that some factor, for example, inhibition of slip at boundaries, is necessary to account for the assumed difference in the behavior of aggregates and single crystals. I have, in fact, obtained some evidence that the latter suggestion may be true for the body-centered cubic lattice for, although it is accepted that iron aggregates readily form Neumann lamellae when subjected to repeated light shocks, I have so far been unable to produce any but slip markings in single crystals of iron when submitted to repeated light impacts.

### B. *Close-Packed Hexagonal Lattice (Zinc):*

Mark, Polanyi and Schmid(96) had shown that under *statical* tensile straining, the principal slip plane of zinc was the basal plane (0001), but their experiments suggested that prismatic planes might also act as slip planes when suitably oriented with reference to the applied shearing forces. To put this suggestion to a critical test, our first fatigue experiment(68) on a zinc crystal was made using a specimen having a specially selected relative orientation of the crystallographic and straining axes. In this specimen, tested under reversed torsional couples, the basal plane of the crystal nearly contained the axis of torsion: as a result, the resolved shear stress on the prismatic planes, *at every point on the specimen*, exceeded the resolved shear stress on the basal plane, and, over a considerable region, was 25 times greater. The experiment yielded very decisive results. The only operative plane was the basal plane, the slip direction being parallel to the principal line of atoms, $[10\bar{1}0]$ type, subjected to the maximum shear stress component; the *distribution* of slip also varied in accordance with the change in the shear stress component and it was clear that the resolved shear stress law was being obeyed exactly. In addition to slip bands, a very great number of mechanical twins were present; the occurrence of these twins was related to the *resolved* *shear stress distribution on the basal plane,* their frequency of occurrence being proportional to the shear stress component although *not* related to the intensity of shear stress or normal stress on the twinning plane itself. Two sets of twins were produced, both of the first order pyramidal (two) type, $(10\bar{1}2)$. In many cases the structure within the twin was identified and it was established that the twinned structure represented a "mirror image" about the twinning plane. Figure 30 shows a typical field where large twins are present. Within the twinned structure itself, slip occurred on the twinned basal plane[1] and many measurements indicated that this slip in the twinned structure was also controlled by the resolved shear stress distribution (it was very interesting to find that the boundary of the twin did not apparently affect the stress distribution). The specimen eventually failed by fatigue, the crack following three main crystallographic directions, being parallel to the traces of the original and twinned basal planes, also, to the trace of the twinning plane. Figure 31 shows a portion of the crack principally following the original and twinned basal planes. In Figure 32, the crack distinctly follows for some distance the edge of a twin.

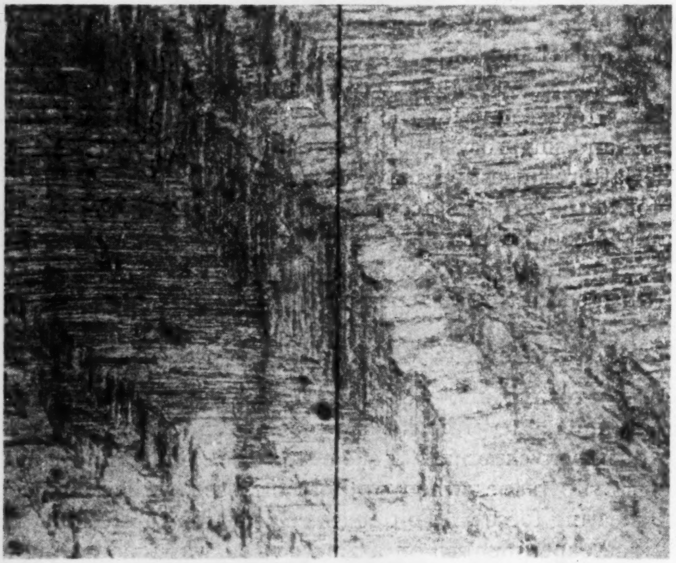

FIG. 30.—Zinc Crystal Showing Large Twins, also Basal Plane Slip in both Primary and Twinned Structure (×330).

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[1] In a primary twin the basal plane makes an angle of 94 deg. 5 min. with the basal plane of the original structure: it was undoubtedly deformation on the twinned basal plane which was confused, by Mark Polanyi and Schmid, with slip on prismatic planes (which are inclined at 90 deg. with the basal plane).

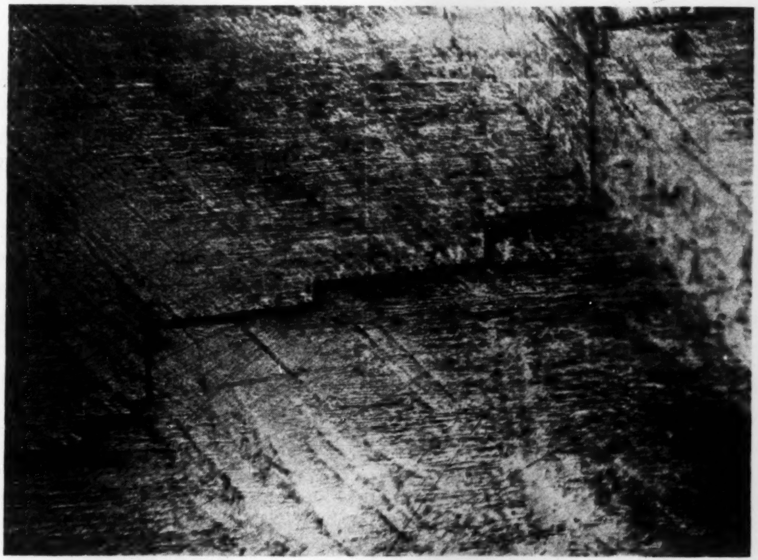

FIG. 31.—Fatigue Crack in Zinc Crystal Along Basal Planes of Primary and Twinned Structures (×100).

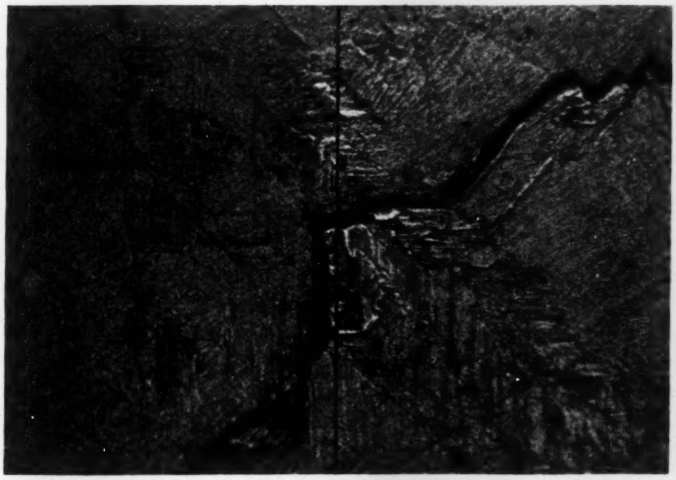

FIG. 32.—Fatigue Crack in Zinc Crystal Following Edge of Twin and Basal Planes (×330).

The fact that the twin distribution was undoubtedly related to the direction of slipping on the basal plane and to the intensity of the shear stress component on that plane led to a very interesting conclusion and prediction. We had found that the production of twins was not related to shear stress intensity on the twinning plane itself, yet the most probable mechanism[1] of twinning would be facilitated by a small shear movement parallel to the twinning plane. Now the rotation of elements on the slip plane arising from slip (refer to Fig. 18) leads to a maximum movement in a plane perpendicular to the slip plane and containing the slip direction. *It appeared probable that the operative twinning planes would be those along which the rotation due to slip on the basal plane produced a component movement.* Figure 33, which sketches the relative positions of the basal and twinning planes, may be helpful in following the argument. In Fig. 33 ($a$) the plane of the paper represents the slip plane, with three possible slip directions $x$, $y$ and $z$. The six twinning planes $T_1$ to $T_6$ thus form 3 pairs of the sides of a regular hexagonal pyramid. Let slip occur on the slip plane in the direction $x$. Then the resulting rotation of the structure will have component movement down the pairs of planes $T_3$ and $T_4$, $T_5$ and $T_6$, but none along the pair $T_1$ and $T_2$. Now, twinning is equivalent to small rotations and translations of atoms in a plane perpendicular to the twinning plane concerned, that is, in the planes whose traces on the hexagonal pyramid of Fig. 33 ($a$) are represented by the lines $OS$. Therefore, in the general case, the rotation due to slip would be expected to produce twins on

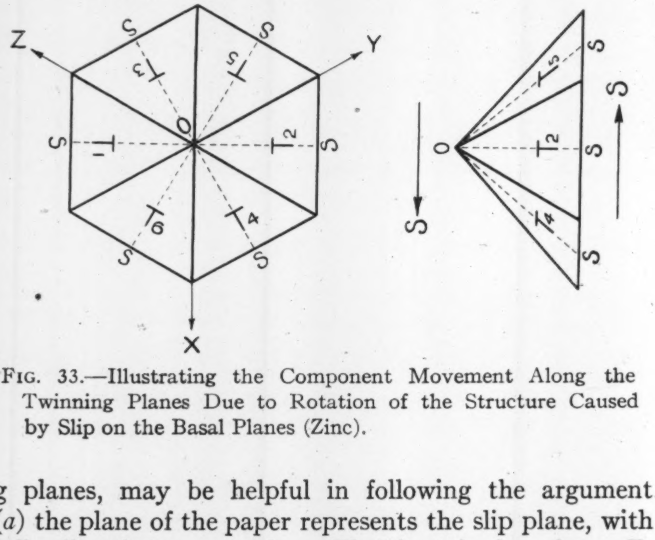

FIG. 33.—Illustrating the Component Movement Along the Twinning Planes Due to Rotation of the Structure Caused by Slip on the Basal Planes (Zinc).

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[1] Our views on this mechanism(68) are in substantial agreement with those advanced by Mathewson and Phillips(100).

those 2 pairs of twinning planes which do *not* contain the direction of slip. This would lead to the possibilities given in Table III. In the first crystal tested under fatigue, only one slip direction became operative and only one pair of twins was produced instead of two pairs as might be expected if no considerations other than shear stress entered into the problem. But in our experiment the stresses were *alternating* and it is conceivable that shear of one sign would produce twins on one pair of twinning planes, according to alternative *A*, say, but the reversal of the shear would not produce twins on the other pair, alternative *B*. To test the prediction, alternating torsion experiments were made on three crystals of the same orientation, so chosen that all three slip directions became operative in different parts of the specimen. We found(69) that, in each case, a total of six pairs of twins was produced, one complementary pair in each slip zone. One crystal conformed to alternative *A* of Table III, while both the other two conformed to alternative *B*; these alternatives are the same sequence with a simple phase difference. *Thus the main prediction, that the operative twinning plane must not contain the slip direction, was fulfilled exactly.* Why only one pair of twins was produced in each slip zone has not yet been determined; suggestions are discussed elsewhere(69). It is believed that these experiments afford the first evidence that mechanical twinning, like slip, can be accounted for *quantitatively*; they offer yet another illustration of the intimate dependence of deformation and failure on the crystalline structure and of the possibilities for the fundamental study of the properties of metals rendered possible by the use of single crystals.

TABLE III.—RELATION BETWEEN OPERATIVE SLIP DIRECTION AND OPERATIVE TWINNING PLANES IN ZINC SINGLE CRYSTALS.

| SLIP DIRECTION | | $x$ | $y$ | $z$ |
|---|---|---|---|---|
| Operative twinning planes | Alternative *A* | $T_3$ and $T_4$ | $T_5$ and $T_6$ | $T_1$ and $T_2$ |
| | Alternative *B* | $T_5$ and $T_6$ | $T_1$ and $T_2$ | $T_3$ and $T_4$ |

### *C. Body-Centered Cubic Lattice (Alpha Iron):*

The mechanism of the deformation of iron is an excellent example of a problem about which no definite information was obtained until the behavior of individual single crystals could be studied in detail. It was well known that "forked" or wavy slip bands were usually produced in this metal and their cause has been the subject of considerable speculation. It had also been shown(57) that straight slip bands in iron sometimes resulted from fatigue stressing. In 1925, Taylor and Elam(142) studied the distortion of iron single crystals under static straining. They found that the distortion occurred by shear, the slip direction being always parallel to a cube diagonal [111] but that the slip plane bore no relation to the crystallographic axes, being determined merely by stress considerations: they concluded that the slip lines had no direct relation with any of the principal crystal planes. To extend this work to the case of fatigue, I made some alternating torsional tests on a single crystal of iron(65).

Three types of slip bands were produced, (*a*) definitely straight, (*b*) two distinct and nearly straight sets of bands of definite slopes which, at their junction, gave a wavy composite band having limiting slopes corresponding to those of the individual bands, and (*c*) definitely wavy bands, with well-defined limits of slope and average slope but impossible to resolve into combinations of straight slip bands. At four positions around the specimen, corresponding to the opposite ends of two diameters, the slope of the slip bands changed abruptly, showing that the slip system altered in some manner probably corresponding to the change in slip plane and slip direction that usually occurs in a metal crystallizing in the face-centered cubic system. Detailed examination showed that the slip direction in each operative slip zone was one of the cube diagonals which represent the directions of closest atomic spacing. The stress analysis was then worked out for what may be termed the "most general case of the resolved shear stress law," that is, that at any point on the surface of the specimen and for a given [111] slip direction, it is required to find the slip plane on which the shear stress resolved in the given direction and along the plane is greater than that on any other of the single infinity of planes which also contain the given slip direction. It may be interesting to compare the equation with the previous equation giving the shear stress on a given *plane* in a given contained direction. Using the same convention as before, the shear stress component becomes

$$S_r = S\sqrt{\cos^2(\psi_d - \lambda)\cos^2\theta_d + \sin^2(\psi_d - \lambda)\cos^2\theta_d}$$

while the coordinates of the plane are determined by the relation:

$$\tan\theta_p = -\cot\theta_d \sec(\psi_d - \psi_p)$$

With the exception that it contains always one of the [111] directions. the slip plane determined in this way bears no relation to the crystallographic axes, changing continuously from point to point around the circumference of the specimen. The stress analysis for the crystal used in the experiment is as plotted in Fig. 34, and was found to account exactly for the change-over points of the slip direction, for the variation in distribution of the slip bands, and for the *average* slope of the slip bands at any point on the surface. The specimen had fractured by the development of 14 fatigue cracks all concentrated around the four peaks of the stress curves shown in Fig. 34. It was thus clear that the fatigue failure of alpha iron was intimately associated with slip and controlled by the same law. Although the distortion was exactly *equivalent* to slip on the planes of maximum resolved shear stress it had obviously not occurred by simple slip *on* those planes. A close study of the shapes and distribution of the slip bands gave results consistent with the following hypothesis:[1]

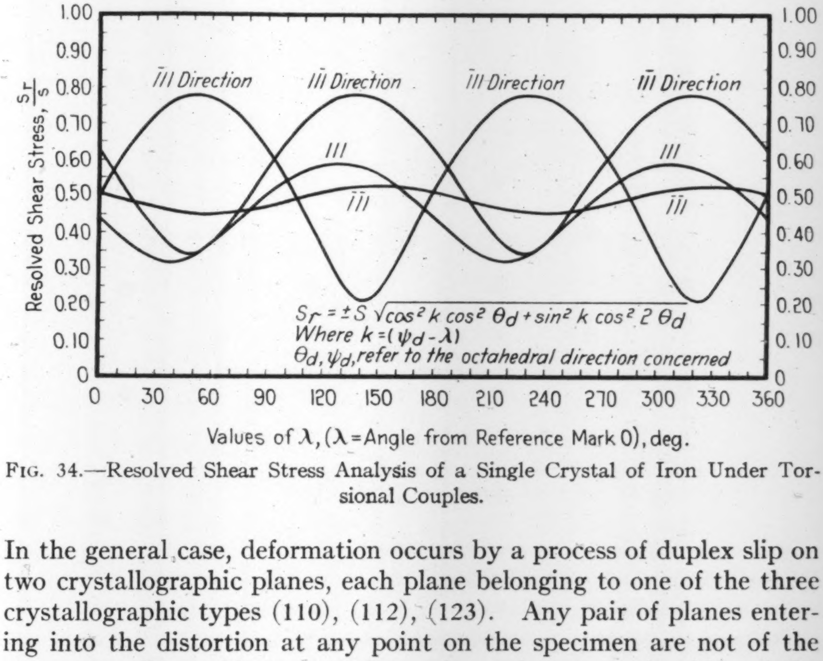

FIG. 34.—Resolved Shear Stress Analysis of a Single Crystal of Iron Under Torsional Couples.

In the general case, deformation occurs by a process of duplex slip on two crystallographic planes, each plane belonging to one of the three crystallographic types (110), (112), (123). Any pair of planes entering into the distortion at any point on the specimen are not of the *same* crystallographic type and are situated on *opposite* sides of the plane of actual maximum resolved shear stress, being determined by the consideration that each slip plane is subjected to a greater shear stress component than any other available slip plane (of the three types mentioned) *situated on the same side* of the plane of maximum resolved shear stress. Owing to the geometry of the structure, slip under these conditions can only occur on one of the two combinations: (*a*) (110) and (123) or (*b*) (112) and (123). Fig. 35 illustrates the suggested mechanism of slip: it shows a view looking through the lattice in a direction parallel to a [111] direction, while *MR*, denotes the plane of maximum resolved shear stress according to the above stress analysis; the relative amounts of slip on the $(\bar{3}12)$ and $(\bar{1}01)$ planes are so adjusted that the resulting deformation is equivalent to a simple shear on the plane *MR*. Fig. 36 shows a field where the components of slip are clearly visible in addition to long composite wavy bands parallel to the plane of equivalent distortion. Figure 37 shows a spot similarly stressed but after greater distortion: the

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[1] This theory is not necessarily in disagreement with the "rod" theory of Taylor and Elam(**142**): it may be regarded as an extension of their hypothesis. On the other hand our observations are definitely not in agreement with the conclusion of Fahrenhorst and Schmid(**32**) that slip is confined to planes of the (123) type.

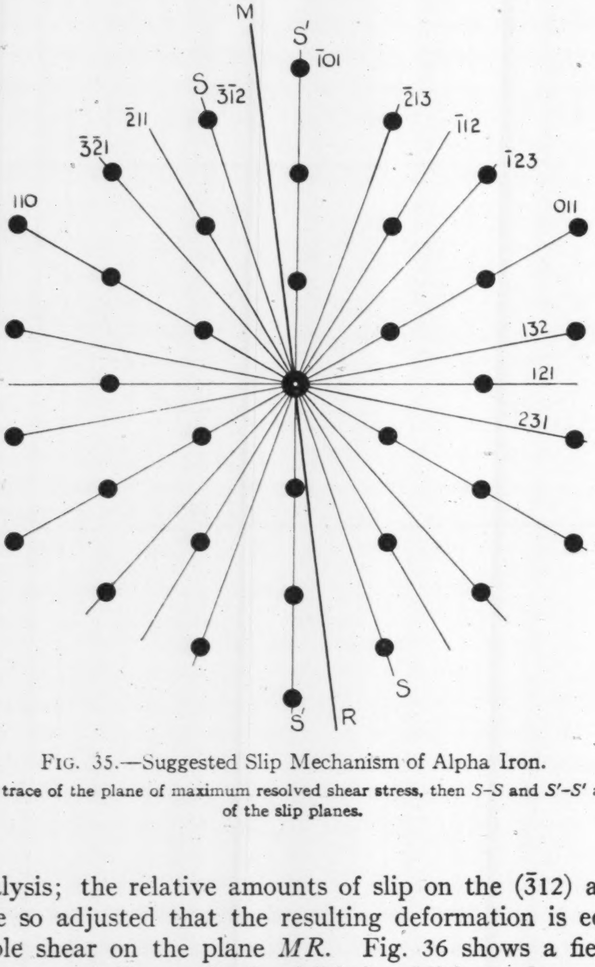

FIG. 35.—Suggested Slip Mechanism of Alpha Iron.

If *M–R* is the trace of the plane of maximum resolved shear stress, then *S–S* and *S′–S′* are the traces of the slip planes.

individual slip components can no longer be detected but have joined together to produce wavy bands of the same average and limiting slopes. Straight slip bands are produced when the slip direction becomes tangential to the surface of the specimen; this *must* occur whatever the type of distortion may be.

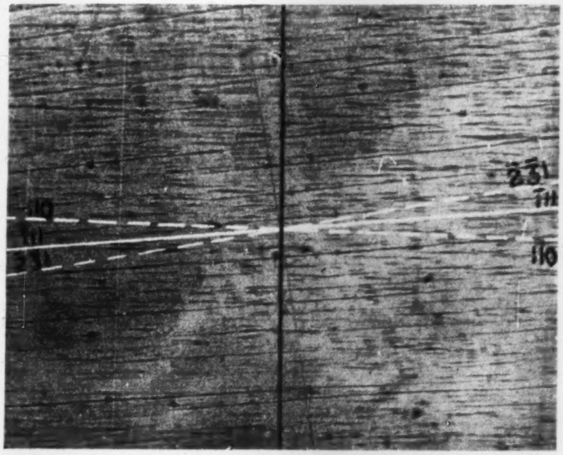

FIG. 36.—Slip Bands in Iron Single Crystal (×100).

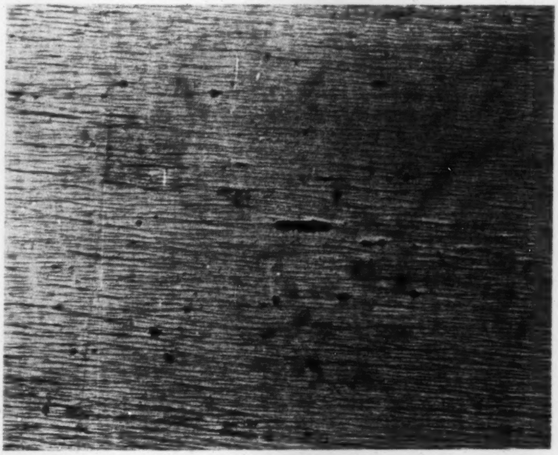

FIG. 37.—Slip Bands as in Fig. 36, but After Greater Distortion (×100).

Fatigue cracks were initiated at the positions of maximum resolved shear stress, but their directions of spread were predominantly axial, suggesting, as in other metals, that the effects of slipping so damage the crystalline structure that cracks formed under torsional fatigue stresses develop along planes of actual maximum, *not* maximum resolved, shear stress. Figures 38 and 39 are typical examples of fatigue cracks in an iron single crystal.

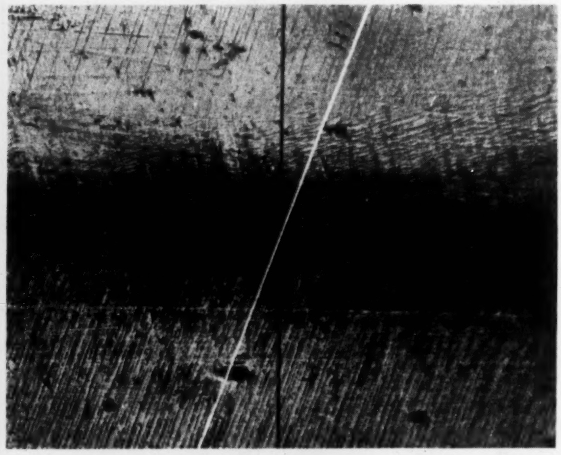

FIG. 38.—Fatigue Crack in a Single Crystal of Iron (×100).

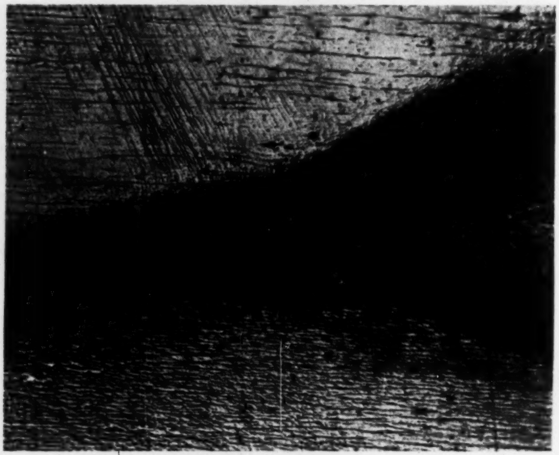

FIG. 39.—Fatigue Crack in a Single Crystal of Iron (×100).

An interesting point about this iron crystal was that it remained elastic during millions of repetitions of a stress range of which the maximum stress component was ±7200 lb. per sq. in. Zinc crystals have exhibited elasticity under stress ranges exceeding ±1100 lb.

per sq. in., while silver crystals exhibited no visible slip bands until the applied range exceeded ±560 lb. per sq. in.; aluminum crystals stressed in fatigue have produced slip bands at lower ranges of stress. These figures are given as an impression appears to have arisen that, in general, metallic single crystals possess no range of primitive elasticity: this is not the case.

*D. Face-Centered Rhombohedral Lattice (Antimony and Bismuth):*

The results of tests on single crystals of iron suggested that the slip and fatigue characteristics were dependent more upon the condition of maximum linear atomic density in the slip *direction* than upon that of maximum atomic density of the slip plane, but the tests on iron crystals were not *critical* in this respect as the (110) planes, which entered largely into the distortion, were also the planes of maximum atomic density (of the body-centered cubic lattice). The face-centered rhombohedral lattice, in which bismuth and antimony crystallize, has the unusual property that the planes of maximum atomic density $(1\bar{1}0)$, $(01\bar{1})$ and $(\bar{1}01)$ do not contain the lines of closest atomic spacing $[1\bar{1}0]$, $[01\bar{1}]$, and $[\bar{1}01]$ all three of which are contained by the (111) plane which is perpendicular to the trigonal axis of symmetry of the lattice. At first sight, it may appear that this type of lattice structure affords a unique opportunity for a critical test on the relative influence of the atomic density of the slip plane and slip direction but this may prove to be fallacious. For the lattice is so complex (see Fig. 1) that the fields of force of the atoms are certainly not spherically symmetrical; simple consideration merely of the interatomic distances cannot present a true view of the interatomic forces. Whatever the result of an experiment on bismuth or antimony, it would be unwise to attempt to apply these results to a simple homogeneous[1] lattice—such as the face-centered or body-centered cubic—where the fields of force of the atoms are probably almost spherically symmetrical, or even to the close-packed hexagonal lattice which is not truly homogeneous. But experiments on bismuth and antimony seemed well worth making and have, in fact, yielded much interesting information, particularly with respect to deformation by twinning.

Two specimens of antimony were tested(70) under torsional fatigue stresses: both failed eventually by cleavage: neither specimen deformed by "slip." A number of fine lines were observed which corresponded to the traces of the (111) plane, which is the plane

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[1] A homogeneous lattice is defined as one in which every atom is similarly situated with regard to every other atom.

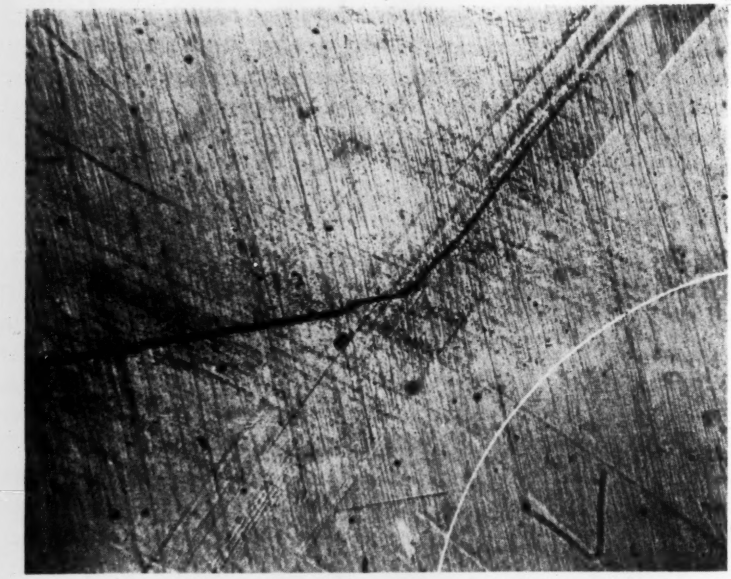


FIG. 40.—Crack in Antimony Single Crystal Formed by Fatigue Stressing (×100).

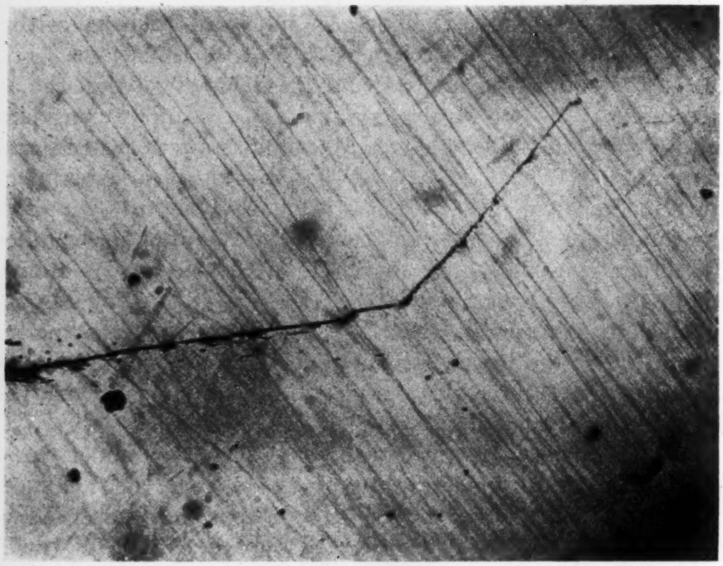


FIG. 41.—Same Crack as in Fig. 40 After a Further 7,000,000 Stress Reversals (×100).

which might reasonably have been expected to act as the slip plane. But the (111) plane was definitely a plane of cleavage and a comparison with cleavage cracks led us to conclude that the fine lines in question were probably incipient cleavages; the distribution of these fine lines agreed very well, however, with the resolved shear stress distribution on the (111) plane. Cleavage also occurred in directions parallel to the three planes (011), (101), (110) which were definitely identified as the twinning planes: these cleavages followed the edges of twins. Of great interest in relation to fatigue was the growth of cracks under the applied cycles of alternating stresses. Figure 40 shows a crack which was observed at a certain stage in the experiment: this crack follows directions parallel to the traces of the (111) plane and one of the twinning planes. After repolishing, the specimen was subjected to a further 7,000,000 reversals of the previous range of stress when this crack appeared as in Fig. 41. It is indeed remarkable, in view of the extreme "fineness" ratio of the crack and of the "brittle" nature of the material, that this crack did not extend appreciably during this lengthy fatigue history; *this observation may be a significant index of the possible danger incurred in applying the theory of elasticity to the finer aspects of the crystalline structure of metals.* A very profuse and beautiful system of twinning resulted from the stressing. The twinning planes were definitely identified as (101), (011), and (110); also, the orientation of secondary twins and secondary cleavages established that the twinned structure was equivalent to a mirror image of the primary structure about the twinning planes. Figures 42 and 43 show typical examples of these twinning effects. No definite evidence was obtained of any relation between the applied stress system, either of shear or normal stress, and the distribution of twins. The cracks and fracture faces followed, in general, well-defined crystallographic planes, either the (111) plane or one of the twinning planes; Fig. 44 is a typical example.

As the experiments on antimony had afforded no evidence of slip characteristics, the experiments were extended(72) to crystals of bismuth, which metal has often been stated to exhibit plastic deformation by slip. Torsional fatigue tests failed, however, to provide any visible slip bands. Profuse twinning occurred which was generally similar to that exhibited by antimony crystals, the twinning planes being identically the same. The distribution of the twins bore a remarkably close relation to the resolved shear stress on the twinning plane and, although it is unlikely that any movement of the nature of slip occurs on planes of such low atomic density as the twinning planes, the evidence suggests that the mechanism of deformation was

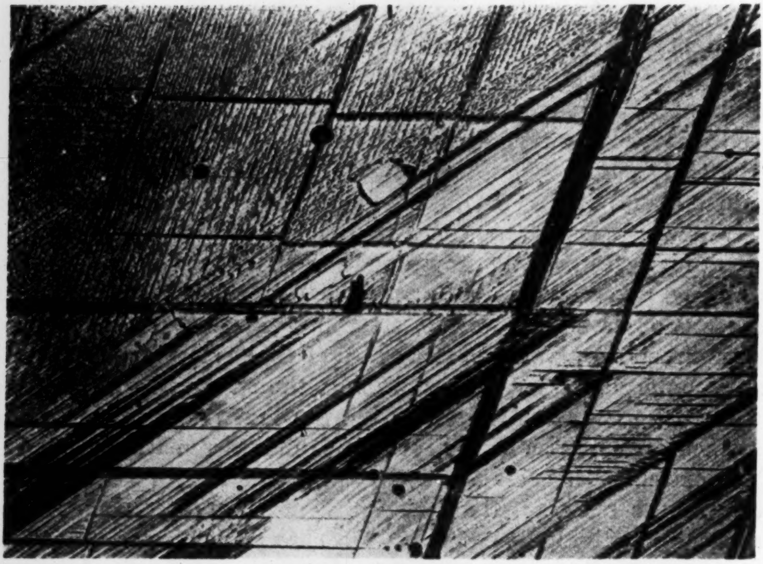


FIG. 42.—Twins Visible on a Cleavage Plane (111) of a Single Crystal of Antimony (×100).

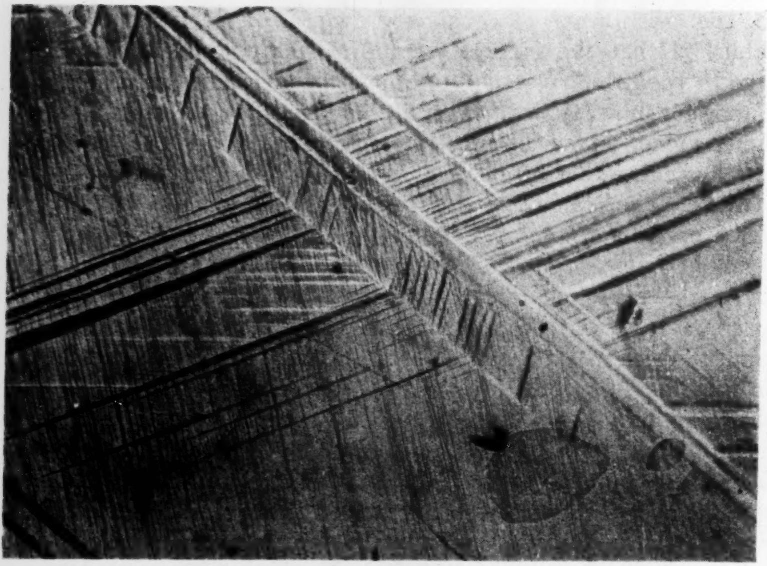


FIG. 43.—Typical Primary and Secondary Twinning in Single Crystal of Antimony (×180).

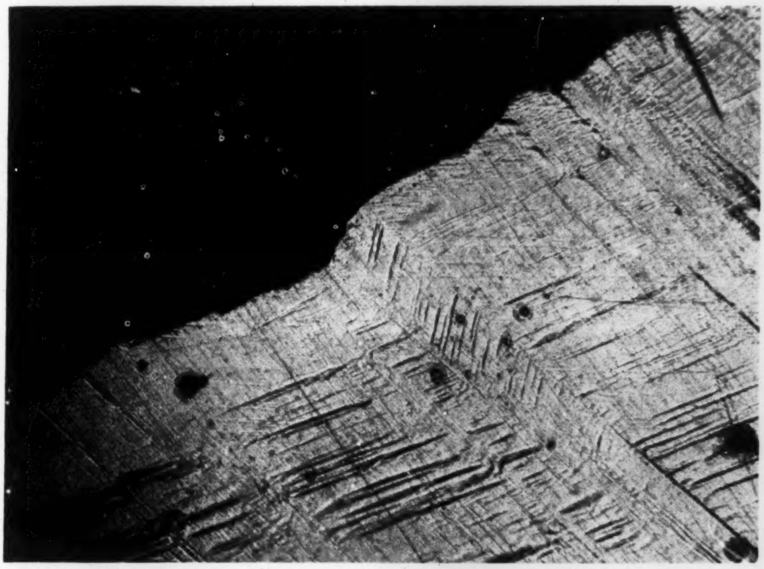


FIG. 44.—Fatigue Fracture of an Antimony Crystal (×100).

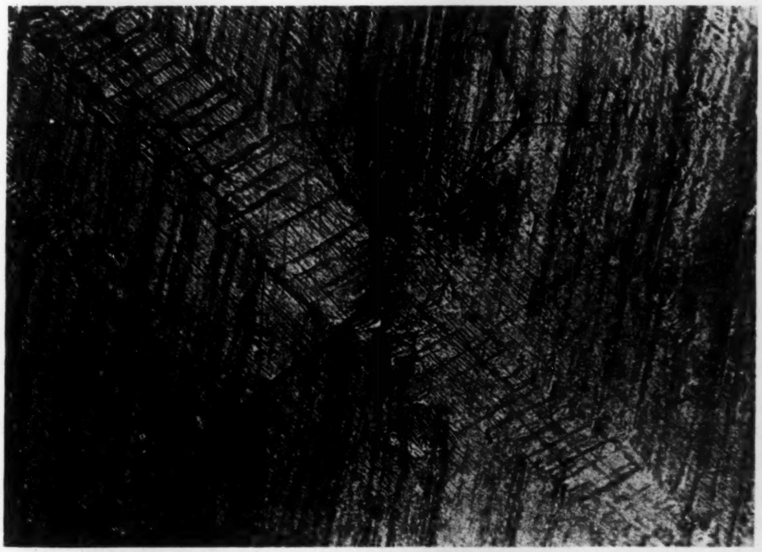


FIG. 45.—Fatigue Crack in a Bismuth Crystal (×180).

one of twinning initiated by partial slip on the twinning plane. As in antimony, the cracks causing failure were in directions parallel to the (111) plane and the twinning planes: a typical fatigue crack is shown in Fig. 45.

Our experiments had thus failed entirely to produce plastic deformation by slip in metals conforming to the face-centered rhombohedral structure, although several references in the literature state that bismuth has a definite slip plane. We had noticed that our own crystals could be bent by hand or distorted plastically (5 per cent extension) in tension, but, in each case, the deformation was accompanied by audible sounds and a mechanism of twinning only was indicated. Georgieff and Schmid(39) had reported that slip occurs provided the shear stress on the octahedral plane is equal to or greater than 0.7 times the normal stress on that plane. To investigate this possibility, we tested a bismuth crystal of the required orientation using cycles of direct stress as, in this way, the maximum change in microstructure would accompany the minimum total distortion of the test specimen. But the only visible features were a large number of twins formed on the twinning plane subjected to the maximum shear stress. From the results of this lengthy series of experiments we concluded that *bismuth deformed plastically, not by slip, but by twinning in the manner described.*

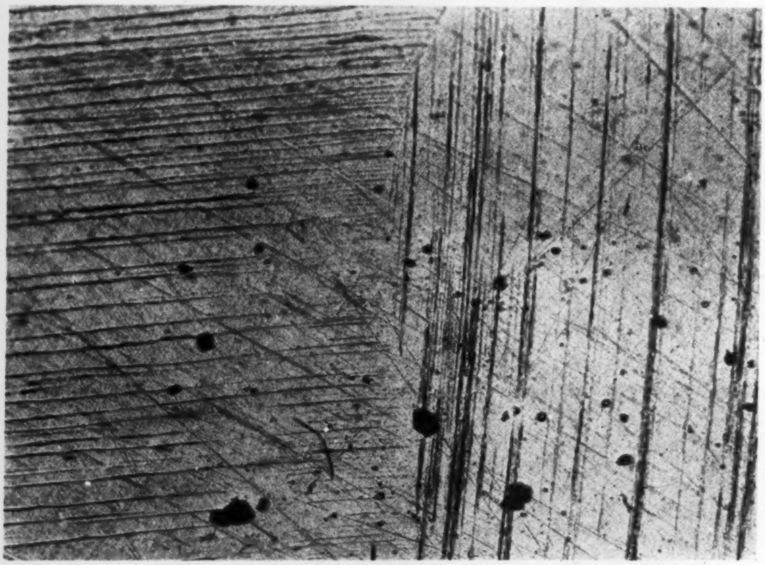
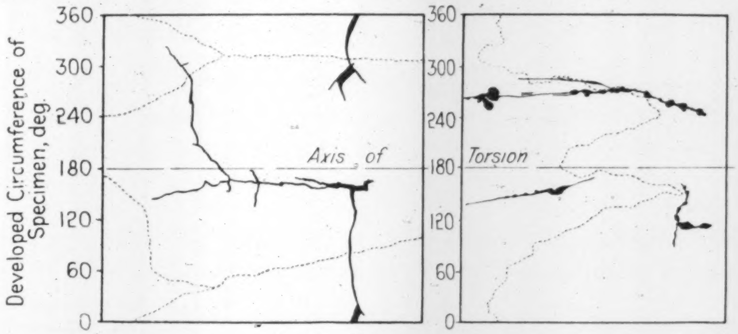


FIG. 46.—Slip Bands at Crystal Boundary (Aluminum) (×180).

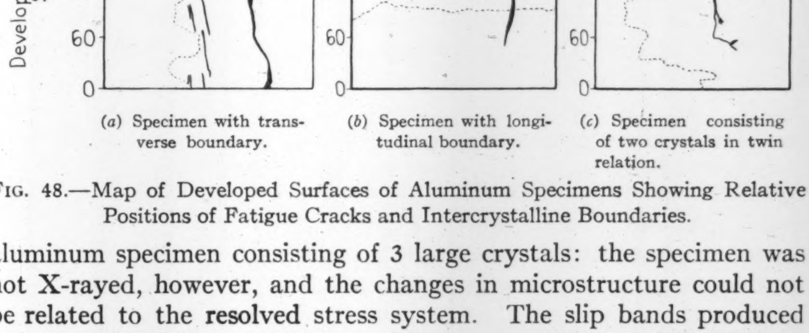
### *E. Influence of the Intercrystalline Boundary on Deformation and Fracture by Fatigue:*

The extension of the investigation of the fatigue research from single crystals to specimens consisting of two or more crystals has only recently been commenced, but some interesting results have already been obtained. A preliminary test (61) was made on an aluminum specimen consisting of 3 large crystals: the specimen was not X-rayed, however, and the changes in microstructure could not be related to the resolved stress system. The slip bands produced were exactly similar to those resulting from similar torsional fatigue tests on single crystals, attended by the same process of gradual strain-hardening under repeated cycles of stress. In many regions, the slip bands extended to the crystal boundaries, changing their


Developed Circumference of Specimen, deg
360
300
240
180
120
60
0
Axis of
Torsion
360
300
240
180
120
60
0

(*a*) Three-crystal specimen tested in air. (*b*) Two-crystal specimen tested in water.

FIG. 47.—Map of Developed Surfaces of Aluminum Specimens Showing Relative Positions of Fatigue Cracks and Intercrystalline Boundaries.


Developed Circumference of Specimen, deg
360
300
240
180
120
60
0
360
300
240
180
120
60
0
Axis of Torsion
360
300
240
180
120
60
0

(*a*) Specimen with transverse boundary. (*b*) Specimen with longitudinal boundary. (*c*) Specimen consisting of two crystals in twin relation.

FIG. 48.—Map of Developed Surfaces of Aluminum Specimens Showing Relative Positions of Fatigue Cracks and Intercrystalline Boundaries.

directions as they crossed; in other regions, the slip bands stopped at appreciable distances from the boundaries which appeared to exert a "shielding" effect on the slip. The cracks formed in the areas of heaviest slip and extended to the boundaries which they crossed nearly orthogonally: no indication of inter-crystalline fracture was found (see Fig. 47 (*a*)). The fatigue strength of the specimen was not appreciably different from that of a single crystal of aluminum, fracture occurring after 31,000,000 cycles of ±2200 lb. per sq. in. nominal shear stress.

Very recently, a thorough investigation(77) has been made of 3 specimens of aluminum, each consisting of 2 large crystals: each specimen was tested to destruction under reversed torsional stresses. In one specimen, the inter-crystalline boundary was mainly transverse to the axis of torsion (Fig. 48 (*a*)); in the second, the boundary was mainly longitudinal (Fig. 48 (*b*)); in the third, the boundary had no particular orientation but the two constituent crystals were in exact twin relation to each other (Fig. 48 (*c*)). The methods of X-ray analysis, stress analysis and microscopical examination adopted for each individual crystal were exactly the same as if the crystal occupied the entire test length of the specimen. With regard to the stress analysis of a 2-crystal specimen of an isotropic material, provided the elastic range is nowhere exceeded, the distribution of stress should be entirely independent of the boundaries; but, even in the case of an isotropic material, this distribution must be altered as soon as plastic deformation occurs in any crystal. A comparison of the distribution of slip bands and fatigue cracks with the stress analysis should therefore afford indications of the extent of the influence of the boundary and yield some information regarding its nature.

In all regions remote from the boundary the slip band distribution in every case conformed to the shear stress analysis. The only difference between the neighborhood of the boundary and elsewhere lay in the size, number, spacing and general appearance of the slip bands. Heavy slip systems tended to continue right up to the boundary while sparser systems tended to stop short. Regions were observed where the spacing of the slip bands decreased in the immediate neighborhood of the boundary: Fig. 46 is a typical example. The process of the initiation and development of fatigue cracks was in all respects exactly similar to that occurring in a single crystal; first, the production of heavy slip in the regions of the maximum stress component, followed by the appearance of a number of fine cracks in these areas, in directions parallel to the traces of the operative slip planes but sometimes propagating along the general directions of maximum nominal shear stress. Nowhere was any tendency evident for the fatigue cracks to develop along the crystal boundaries; in some cases, smaller cracks were developed in regions very close to the boundary but their course is illustrated by a typical crack which, starting in one crystal, crossed the boundary and subsequently re-crossed twice. As the general direction of the fatigue cracks was in all cases transverse to the axis of torsion, the effect of the boundary on the actual fatigue strength was not directly ascertained. For, in the specimen having the boundary generally transverse to the axis, fatigue cracking was principally confined to the crystal most unfavorably oriented, and the specimen was substantially equivalent, in its last stages, to a single crystal, the progress of the main crack not being affected by being forced to cross a boundary. In the specimen having a longitudinal boundary, however, the spreading crack encountered a boundary directly in its path. As no crystal was tested to destruction under a constant range of stress, no accurate determination of their fatigue limits was obtained, but that of the first crystal was certainly less than ±2000 lb. per sq. in., while that of the second crystal was probably not much lower than ±2900 lb. per sq. in. Further tests are in hand to determine the influence of the *orientation*[1] of the crystal with respect to the axis of torsion and to the crystalline boundary and, until these results are available, the above comparative figures should be discussed with some reserve. While it is quite certain that the slip band distribution was controlled entirely by the values of the shear stress components which ignored the presence of the boundaries, yet the different behavior of the two specimens with regard to the rate of exhaustion of slip and of the production of visible cracks showed that, in some manner, the influence of the boundary was considerable. Two explanations are offered. The effect may lie in some *restriction* of strain which may act not so much by the limitation of the *occurrence* of slip as by the partial prevention of the *damage* due to slip: it is proposed to investigate this aspect using X-ray methods. The second possibility is that the *incidence* of the cracking stage may have occurred in both crystals after similar fatigue histories but that the propagation of the submicroscopic cracks into cracks of visible dimensions may have depended on the presence or absence of boundaries in the direct paths of the cracks. Future research may throw light on these explanations or offer other possibilities. It may be found that the known behavior of the aggregate (failure by "local" fatigue, strengthening with decreasing grain size, etc.) may be explicable on two simple lines—the effect of varying orientation and the resistance offered by grain boundaries to the propagation of fatigue cracks.

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[1] See work of Fahrenhorst, Matthaes and Schmid(30,31).

Particular interest attaches to the fatigue behavior of the twin specimen, of which the two constituent crystals had one octahedral plane in common. In one crystal this octahedral plane was the operative slip plane over a considerable region, while in the adjacent crystal the shear stress on this plane, although high, was exceeded by that on another octahedral plane, the minimum actual difference in stress being only about 2 per cent. It was absorbingly interesting to ascertain whether slip would extend through the boundary on the common plane, thus disregarding the difference in shear stress. Right up to the stage of final fracture, however, the slip distribution exactly obeyed the resolved shear stress law, the operative slip plane changing at the boundary from one plane to another; a really remarkable verification of the accurate response in behavior of a crystal to a simple stress criterion. This specimen also furnished another very interesting feature. Two cases were observed in which "outcrops" of one crystal occurred through its neighbor, and in both, the slip band distribution was exactly as in the parent crystal. Figure 49 shows one of these cases where slip is visible in both crystals and in

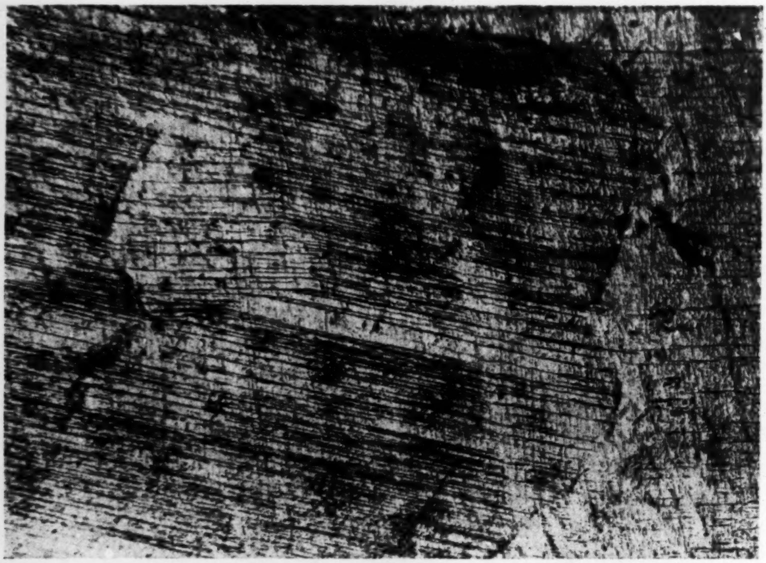

FIG. 49.—Slip Band Distribution in an "Outcrop" and at a Boundary, Due to Fatigue (×100).

the outcrop. This observation suggests that in a finely-divided crystalline aggregate the slip distribution and fatigue cracking may still follow the resolved shear stress law, thus affording hope that the behavior of the aggregate will be directly related to that of the large single crystal.

The fatigue cracks in these four specimens, in relation to the traces of the intercrystalline boundaries, on the surface, are shown in Figs. 47 and 48.

Very little quantitative data is at present available regarding the relative fatigue strengths of the aggregate and single crystal. Comparative fatigue tests(63,64) have been made on single crystals of aluminum and on the aluminum aggregate from which the crystals were prepared. The aggregate was tested in two conditions: (*a*) as hot rolled, (*b*) hot rolled, followed by identically the same *annealing* history[1] as used in the production of the single crystals. The fatigue limits, under cycles of reversed direct stress, of the three materials, were, approximately, as follows:

| | |
|---|---|
| Hot-rolled aggregate | ±7400 lb. per sq. in. |
| Annealed aggregate | ±4900 lb. per sq. in. |
| Single crystals | ±3350 lb. per sq. in. |

These values, however, are based only on the range of applied load in terms of the original area of the specimens: by ignoring the change in cross-sectional area occurring during the test they lose all real comparative value. For the deformations were in the order: single crystal > annealed aggregate > hot-rolled aggregate. Much greater interest attaches to the very accurate determinations of the changes in density which took place during the fatigue stressing. The density of the material in the form of the aggregate definitely decreased by amounts varying from 0.037 to 0.100 per cent of the original value, while that of the single crystals remained unchanged. These results, which are in agreement with results obtained by other workers(103) using *static* stressing, suggest that the decrease in density of metals due to cold work marks an effect which is confined to the neighborhood of crystal boundaries; where no such boundaries are present, as in single crystals, changes of density do not occur.

### *F. Corrosion-Fatigue in Relation to Crystalline Structure:*

Although the important subject of corrosion-fatigue has received intensive study during the last decade—in this connection the valuable work of McAdam is outstanding—no systematic study has yet been made of the changes in microstructure of metals produced under

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[1] But without, of course, the preceding critical strain which led to the production of the monocrystalline state.

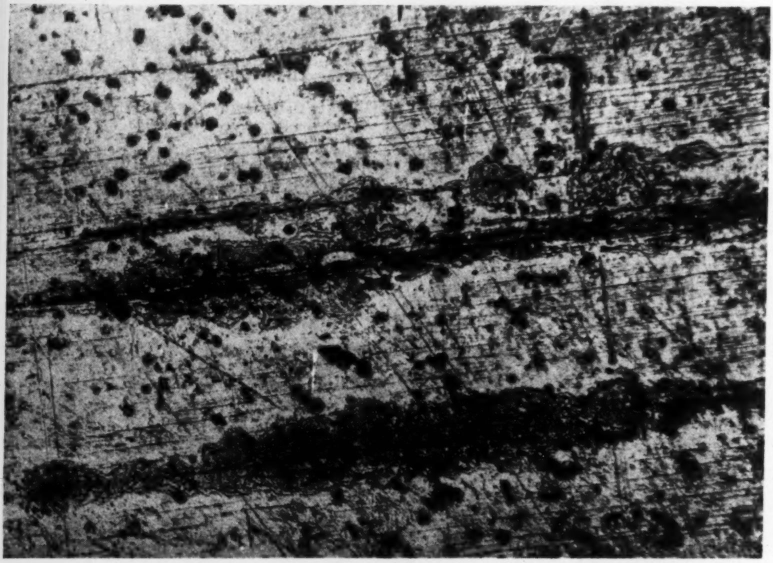

FIG. 50.—Corrosion-Fatigue of a Single Crystal of Aluminum Showing Selective Corrosive Attack and Initiation of Cracks Along Slip Bands (×100).

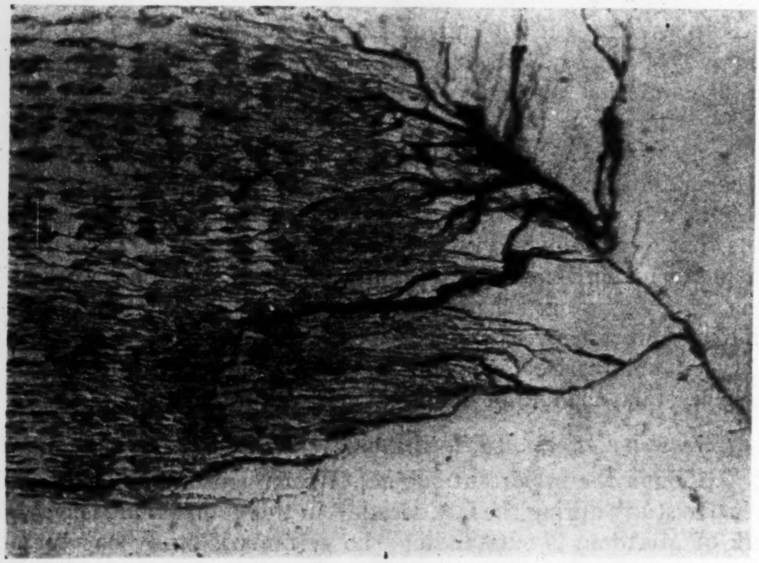

FIG. 51.—Mesh of Fine Cracks at End of Corrosion-Fatigue Crack Within the Interior of an Aluminum Single Crystal (×330).

these conditions. In a survey(75) of the subject, I pointed out that little information was available on such primary aspects as the location of the initiation of a corrosion-fatigue crack, also, whether the normal cause of a crack was trans- or inter-crystalline, etc. The use of single crystals and large-crystal specimens offers unique opportunities of investigation in this respect, as the observed effects can be directly correlated with the applied stressing system and the crystalline structure; also, the results obtained should be useful when studied in relation to the mechanism of deformation and fatigue obtaining under ordinary atmospheric conditions. Some experiments on these lines were, therefore, commenced at the National Physical Laboratory, and two interesting results are now available.

A single crystal of aluminum was tested(71) to destruction under a constant range of alternating torsional stresses, the specimen being immersed in a slow-running stream of tap water. The applied alternating maximum range of resolved shear stress was ±1350 lb. per sq. in.; this would not have produced fracture under atmospheric conditions, but complete failure occurred after 23,700,000 stress cycles. The slip band distribution was strictly in accordance with the resolved shear stress analysis and strain-hardening of the crystal under repeated stressing took place as in previous tests; no fresh slip bands appeared after the initial stage of the test. A general corrosion attack resulted in masses of small pits of which the measured initial occurrence was about one million to the square inch: the distribution of these pits was perfectly general, not being related to the stress conditions.[1] The essential cause of failure was the marked preferential corrosion that took place on the site of the slip bands: due to this cause, between 40 and 50 large cracks, also, a multitude of small cracks, were produced. The larger cracks were all parallel to the traces of the operative slip planes and changed direction with the change of operative slip plane; Fig. 50 is a typical field showing cracks in early stages of development. A particularly interesting feature was the manner in which some of the cracks, after penetrating to a considerable depth into the specimen, suddenly opened out into an intricate mesh of fine cracks of which Fig. 51 forms an excellent example. Under atmospheric conditions, the process of fatigue is directly associated with those areas which have been cold worked by slip; the same correspondence is now proved to exist under corrosion-fatigue conditions. I suggest that this experiment is of very great assistance in interpreting some characteristics of the corrosion-fatigue of metallic aggregates. It offers an explanation why cold-working, although raising the fatigue resistance of a metal, renders the material particularly liable to corrosion-fatigue. Again, certain forms of heat treatment involve the setting up of severe internal stresses, although increased tensile strength and fatigue resistance follow. When such a metal is tested under corrosion-fatigue conditions, its range of primitive elasticity is very small; slip then occurs with the accompanying destructive effects shown so clearly in the single crystal experiment.

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[1] A subsequent experiment(74) showed that similar general pitting is produced under conditions of *stressless* corrosion, also that the fatigue resistance of a crystal is not appreciably reduced by the presence of these pits.

A similar experiment(78) has recently been made on an aluminum specimen consisting of two large crystals with the intervening boundary. A constant range of alternating torsional couples, again producing a range of resolved shear stress of ±1350 lb. per sq. in., was applied throughout; the specimen developed visible cracks after 3,000,000 stress cycles, and these propagated slowly until a total of 29,000,000 stress cycles had been endured when the test was discontinued. All the changes in microstructure were correlated in the usual manner to the stress analysis and the crystalline structure, using X-ray analysis. The slip band distribution showed the usual agreement with the shear stress analysis, and the process of preferential corrosion along slip bands with subsequent development into fatigue cracks was exactly similar to that observed in the single crystal experiment. The chief present interest of the experiment lies in the initiation and propagation of the cracks with regard to the intercrystalline boundary: see Fig. 47 (*b*). These cracks started in a highly stressed portion of a crystal and spread in directions parallel to the traces of the operative slip planes. Arriving at the intercrystalline boundary, the crack crossed this and continued its progress into the neighboring crystal parallel to the traces of the operative slip plane of this crystal. In a region where the shape of the boundary was very irregular, one crack crossed the boundary no less than three times, but *there was no evidence whatever of any tendency of a crack to follow the boundary; neither was there the slightest evidence of any corrosion attack along the boundary.* The only effect the boundary appeared to exert was a slowing-down of the rate of the propagation of the cracks in its neighborhood.

These experiments have, therefore, shown that the process of corrosion-fatigue, like ordinary fatigue and deformation occurring under static and impulsive forces, becomes intelligible when studied in relation to the crystalline structure; also, additional evidence is afforded of the very close connection existing between slip and both types of fatigue failure.

*G. Some Conclusions Regarding the Mechanism of Fatigue Failure:*

In reviewing the experiments described above and endeavoring to apply the lessons learned to the general problem of fatigue as encountered in engineering materials, a most significant result has emerged from the tests made on specimens consisting of two and three large crystals, namely, that *the essential characteristics of deformation and failure of these specimens are substantially the same as observed in the failure of monocrystalline specimens.* The effect of orientation is probably appreciable, the presence of the intercrystalline boundary to some extent inhibits slip, it also appears to restrict the "damage" caused by slip, and it may influence the rate of propagation of fatigue cracks. But these are merely differences of *degree*, classifiable as modifying influences. No essential difference in the *nature* of fatigue failure has been remarked between single crystals and polycrystalline aggregates. Therefore, I suggest that the general problem of fatigue is concentrated in the fatigue failure of the single crystal; if the latter problem can be solved in a fundamental manner, its extension to the case of the polycrystalline aggregate should be relatively simple. Considering, then, the fatigue characteristics of a single crystal of a *ductile* metal, it is undeniable that fatigue failure is inseparably associated with failure of elasticity by the process of slip. I deliberately use the term "associated," because it is certain that failure of primitive elasticity and the initiation of fatigue failure are not simultaneous events; although the latter occurs when the applied external forces produce ranges of strain which exceed the range of the "natural" or acquired elastic limits of the metal, we have shown conclusively that no constant relationship exists between those primitive and natural ranges of elasticity. On the other hand, it is certain that failure in both cases is determined almost entirely by the criterion of resolved shear stress, normal stress being apparently only of minor importance. Also, the initial location of failure is common to both, being always concerned with plastic movements along certain crystallographic directions and, in the general case, parallel to certain crystallographic planes. *Fatigue failure must, therefore, be considered as a consequence of slip; if this conclusion is accepted, the subsequent stages of fatigue become comprehensible in the light of observed phenomena.* The primary problem of the real nature of slip remains, of course, unsolved, but an important advance has been really achieved in the study of the *fatigue* problem if it can be accepted that failure by fatigue is merely a special case of failure under all forms of stressing actions, including that by static and impulsive forces. At one time, it was suggested that fatigue failure was a special problem of its own, but I believe that our work on crystals can be confidently claimed to have shown that this is not the case. Accepting that the property of the plastic deformation of metals by slip is the *primary* problem, which will no doubt be solved at some future time, I suggest that the subsequent failure under fatigue stressing can be interpreted in a fairly satisfactory manner. It is evident that the result of slip is not a mere relative translation of adjacent parts of the crystalline structure: X-ray analysis has proved, that the structure becomes fragmented into component parts of differing orientation. In some metals, the inner structure of these fragments exhibits measurable lattice distortion, but this is not always the case and, hence, cannot be an essential factor in fatigue. Again, the marked strain-hardening accompanying slip is not a direct consequence of fragmentation, for reasons previously stated. Also, it is clear that the initiation of fatigue failure does not mark the attainment of a maximum value of strain-hardening of the crystal *as a whole*, for our experiments have shown definitely that, viewed macroscopically, the hardness due to cyclic stressing increases with increasing range of stress far beyond the stage at which fatigue cracking occurs. I conclude, therefore, that fatigue failure does not result from the consequences of slip and strain-hardening on the crystalline structure in general but only in certain local regions, these regions being those of the junctions of the crystallites formed by crystal break-up. At these junctions some lattice bonds will be in a state of severe strain and, as the process of cold-working by repeated stressing is continued, the limiting lattice strain of some of these bonds will be exceeded, producing rupture and the formation of discontinuities in the lattice. That the average value of the applied external forces which can produce fatigue is small does not affect the argument, for the action is so local that the local and average strains might be of entirely different orders of magnitude; for similar reasons, X-ray analysis is not sufficiently precise to disclose the presence of such local lattice distortions or gaps in the crystalline structure. Provided the applied external strains do not exceed a certain limiting value, at some stage a state of equilibrium is attained which is marked by the cessation of plastic deformation, the material exhibiting a condition of quasi-elasticity involving strain hysteresis. This characteristic of strain-hysteresis under repeated cycles of stress is attributed(59,66) to the movements of certain atoms situated at the junctions of crystallites, these atoms possessing alternative positions of stability with respect to the structure of the neighboring crystal fragments;[1] cyclical movements of this type are consistent with the condition of elastic hysteresis in which a crystal is capable of transforming strain-energy, derived from the external straining system, into thermal energy without involving cumulative damage to the crystal. Such are the conditions obtaining under repeated applications of a range of stress which, although involving plastic deformation, strain-hardening, crystal fragmentation, etc., do not lead to the initiation of visible fatigue cracks and ultimate fracture. When cycles of a range of stress exceeding the "fatigue limit" are applied, essentially similar consequences follow with one important difference, that the relative orientation of neighboring crystal fragments exceeds that value at which truly cyclic conditions can be established. The rupture of atomic bonds then becomes a cumulative effect, with the result that the discontinuities of structure develop through the stage of submicroscopic cracks into that of visible cracks which spread, under repeated stress cycles, in the well-known form of the creeping fatigue crack.[1] The suggested mechanism of fatigue is consistent with the proved initiation of failure in the regions of previous heavy slip. Also, as the discontinuities of structure are not necessarily associated with those crystallographic planes on which slip occurred, it would not be expected that the path of propagation of the cracks should be related to the stress conditions controlling slip; normal stresses might be a powerful factor in influencing the rate of propagation of fatigue cracks and this has been found(132,79) to be the case. Again, the local areas of marked lattice distortion would be susceptible to corrosive influences and the results of the corrosion-fatigue experiments on single-crystal(71,74) and two-crystal specimens(76) afford striking evidence of the general application of the theory. I suggest that the above conception of fatigue as a *consequence* of slip appears to be satisfactory as far as ductile crystals are concerned.[2]

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[1] This conception of the cause and nature of hysteresis was first stated(59) by us in 1924 and arose from our study of the stress-strain characteristics of aluminum: the theory of Prandtl(27) (1928) appears to be very similar.

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[1] The difference in behavior of specimens subjected to ranges of stress just inferior to, and just exceeding, the fatigue limit will not, therefore, be sufficiently marked to warrant the hope that a successful method can be devised of determining the fatigue limit by "shortened" methods (strain, rise of temperature, etc.); before the finer aspects of fatigue had been established by tests on single crystals, such methods(55,56) offered promise.

[2] Many aspects of fatigue phenomena offer tempting opportunities for discussion in this connection but space does not permit. One particular aspect, however, is so fundamental that brief passing reference must be made: I refer to the interesting problem of whether time, as such, enters into fatigue phenomena. Using single crystals subjected to repeated cycles of a safe range of stress, we have established that the process of strain-hardening is not completed during a single loading but many thousands of stress cycles may be required before a cyclical condition is fully established with clearly defined natural limits of elasticity. This extended history does not *necessarily* mean that time is involved for one can reasonably visualize that any one loading operation results in a state of the structure (internal stresses, etc.) such that further deformation occurs when the loading is removed or reversed. Therefore, viscous forces are not necessarily in operation. Yet, if the fatigue limits of a material are determined over a wide range of cyclic frequencies, Jenkin has shown that the fatigue limit increases with increasing frequency; although the effect is small up to values of about 3000 cycles per minute it rises rapidly at higher speeds. Is this an effect of speed on fatigue? I suggest that this may not be the case, but rather an effect of speed on *slip*. With one of my colleagues, I am taking load-strain diagrams of metals subjected to failure under impacts of various velocities of fracture. It is already clear that as the velocity rises, the yield point tends to be suppressed accompanied by an increase in breaking load. This is clearly a speed effect on plastic deformation where viscous forces, for some reason as yet unknown, are in operation. The hypothesis that fatigue failure is a consequence of crystal break-up due to slip at once suggests the explanation of this effect of cyclic frequency on the fatigue limit. If the plastic deformation is reduced, an arrest or delay of fatigue failure would be expected. I believe this aspect has not received previous consideration and I should welcome the views of those interested on the proposal.

It is, in the present state of our knowledge, extremely difficult to visualize this condition of the structure after slip has occurred, as much more quantitative X-ray data is required. It may, however, be of some assistance, in connection with the tentative ideas expressed above, to indicate, in a purely diagrammatic manner, a condition which *may* be consistent with the present evidence and Fig. 52 is offered, with considerable hesitation, for this purpose. In this diagram, the plane of the paper includes the slip direction and is normal to the slip plane. After slip and crystal break-up have occurred, X-ray evidence suggests that the slip planes have become permanently distorted in some manner generally similar to that shown in Fig. 52 (*a*). This might be interpreted as equivalent, in those metals which exhibit measurable lattice distortion after plastic deformation has occurred, to the state of affairs sketched in Fig. 52 (*b*), but it is extremely unlikely to represent the general case and, almost certainly, does *not* indicate the condition of aluminum and similar metals, where no measurable lattice distortion can be detected. Therefore, it appears more probable that the structure is in a condition more akin to that as sketched in Fig. 52 (*c*). In this sketch, the areas marked *A*, *B*, *C*, *D* and *E* represent crystallites free from measurable lattice distortion, but displaced

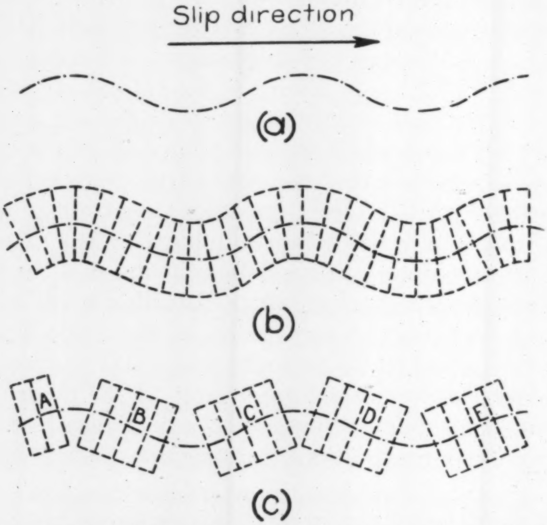

FIG. 52.—Diagrammatic Representation of Possible Relation Between Effects of "Crystal Break-up" and Strain Hysteresis and Fracture Under Static or Fatigue Straining.

relative to each other and to the original average orientation of the structure before slip has occurred. Between these crystallites presumably exist areas in which some lattice bonds are subjected to severe internal strains, while actual discontinuities may occur in certain regions. It is suggested that the structure in such regions is responsible for the observed phenomenon of strain hysteresis; the formation of the submicroscopic crack, which leads to fracture under static or fatigue conditions when the applied strains are of sufficient magnitude, are also suggested as having their location in these regions.

With regard to the fatigue failure of "brittle" metals, the available evidence is extremely limited and one hesitates in drawing general conclusions: the only available data of which I am aware relates to the small number of tests we have made on antimony and bismuth. These metals did not deform plastically and consideration of the consequences of slip is, therefore, excluded. The most common method of failure of these crystals, under repeated cycles of stress, was by direct cleavage on clearly defined crystallographic planes. In these cases the cracks may have been present before the alternating stresses were applied in which case their propagation by alternating stresses was the observed action. Cases, however, were certainly observed of cracks spreading along the edges of twins (Figs. 41 and 45); the atomic structure cannot be regular along these twin edges. If the fatigue cracks were initiated due to such discontinuities of structure, then fatigue in both ductile and brittle crystals may be regarded as due to the same essential cause, the difference merely being that in the ductile crystals, the formation of the discontinuity is a consequence of slip while, in the brittle crystals, the discontinuities are present initially in an imperfect structure. It may be that the fatigue failure of zinc crystals, where the cracks follow the traces of the slip plane *and* the twinning plane (see Fig. 32), forms an excellent example of a metal where both actions can be observed in simultaneous operation. It will, at any rate, be granted that the fatigue characteristics observed in antimony and bismuth crystals do not throw any doubt on the above working hypothesis offered to account for the fatigue failure of ductile metals.

## IX. COMPARATIVE BEHAVIOR OF SINGLE CRYSTALS AND POLYCRYSTALLINE AGGREGATES

Much further research is required before an adequate comparison can be made of the stress criteria governing the behavior of the single crystal and the polycrystalline aggregate, but the present position is of sufficient interest to warrant brief discussion. For purposes of comparison, the stress conditions which cause failure of primitive elasticity are unlikely to provide fruitful information, principally because of the experimental difficulties involved in the determination of the value of the elastic limit: in fact, the values obtained seem to be more directly related to the *accuracy* of the strain measurements than to any real physical property of the material, whether monocrystalline or polycrystalline. Also, the stress conditions at fracture are extremely complex and are probably incapable of exact determination. There remain the conditions determining plastic flow and the fatigue limit, of which only the first named has as yet received detailed experimental attention.

For the stress criterion determining the conditions of plastic flow in polycrystalline aggregates, several different forms have been suggested. At the present time, it appears to be generally agreed that the criterion must be chiefly related to shear stress[1] and the problem is concerned with the form[2] of the appropriate expression. If $p_1$, $p_2$, and $p_3$ $(p_1 > p_2 > p_3)$ denote the values of the three principal stresses, then Mohr's theory states that plastic flow occurs on the planes of maximum shear stress but is also influenced by the value of the normal stress on these planes; thus, the stress criteria involve $(p_1 - p_3)$ also $(p_1 + p_3)$. The alternative hypothesis, due to von Mises,[3] states that the criterion for plastic flow is determined by the condition that the expression

$$\left\{ (p_1^2 - p_2^2) + (p_2^2 - p_3^2) + (p_3^2 - p_1^2) \right\}$$

shall have a constant value. The investigation of the correctness of the hypothesis of Mohr or von Mises when applied to the plastic yielding of polycrystalline aggregates has received detailed experimental attention by Lodé(93), also Taylor and Quinney(145); in both cases, the applied stress conditions were such as to investigate a great variety of ratios of the principal *shear* stresses and thus to explore fully the influence of the intermediate principal stress; this influence marks the chief point of difference between the two theories under discussion. Lodé's results were sufficient to show conclusively that Mohr's hypothesis was *not* fulfilled, and afforded important experimental confirmation of the von Mises *stress* criterion. But the von Mises hypothesis also requires that, as in the case of a viscous fluid, the ratio of any pair of the principal shear stresses to the ratio of the principal shear strains shall be equal to one another; this relation, termed the second law of von Mises by Taylor and Quinney, was investigated fully by these investigators in addition to the stress criterion for yielding. With regard to the stress criterion, they found that the von Mises relation was followed closely by copper and aluminum, but not so closely by mild steel (the discrepancy was ascribed to lack of isotropy due to a coarsened grain size) but even in the latter case, the results were in much closer agreement with the von Mises relation than with that of Mohr. From the second law of von Mises, the results showed regular and consistent deviations; results obtained from heated glass agreed exactly with the law. Summing up, it may be concluded that the stress criterion for the yielding of polycrystalline aggregates conforms very closely to the von Mises relation: the Mohr hypothesis is not verified.

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[1] It is regarded as established that the application of hydrostatic pressure, either to a single crystal or an aggregate, does not cause plastic deformation.

[2] The simplest form, usually known as Guest's law, has not been found consistent with the results of experiment: this theory states that plastic yielding is determined solely by the maximum value of the shear stress (greatest difference of the principal stresses).

[3] Although this hypothesis was suggested by von Mises to overcome certain mathematical difficulties in the *form* of a suitable criterion, it has since been shown, by Hencky, that the von Mises expression has a definite physical meaning, being a measure of the potential energy stored in the material due to the purely elastic deformational (shear) strains, excluding uniform contraction or expansion.

Considering the deformation of single crystals, we have seen that plastic deformation and fatigue failure are both controlled essentially by the simple criterion of the greatest shear stress component resolved on certain planes and contained directions. The fact that normal stress on the slip plane has no discernible influence on the deformation is sufficient to disprove the applicability of the theory of Mohr; the question remains whether the plastic yielding of polycrystalline aggregates according to the von Mises hypothesis is consistent with the criterion of resolved shear stress for the single crystal. Now if the plastic deformation of an aggregate is determined entirely by the mechanism of deformation of the individual crystals, the effect of the boundary being neglected, then the stress criterion for the former would be expected to lie between the extremes defined by the application of the resolved shear stress law to all possible orientations of the crystals. That is to say, the average yielding load of the aggregate should represent the average value of the yielding loads of the crystals, the latter value being determined by the type of the applied forces and by the type of the crystalline structure. As far as I am aware, the required statistical analysis for the general case has not yet been carried out. To obtain some indication of the general result, however, the case has been worked out[1] of the face-centered cubic lattice under the stress systems of simple tension and pure shear. It is found that the ratio of the critical stress in tension (in the aggregate) to the critical resolved shear stress (in the single crystal) has the value of 2.23, while for torsion the value is 1.13. Thus, if a purely "orientation" effect obtains, the ratio of the tensile yielding stress of an aggregate to the stress at yielding in torsion, for this particular lattice structure, should have the value 0.507; the generalized form of the von Mises relation gives the corresponding value of 0.577. The result is, of course, mainly of a negative nature; it demonstrates that the von Mises' hypothesis does not represent a statistical generalization of the single crystal criterion, but it affords no indication of the criterion which statistical generalization would establish; the nature of the analysis by which the quoted results were obtained did, however, suggest that the generalized criterion would not be simple in form.

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[1] I am indebted to my colleagues, Cox and Sopwith, for these results.

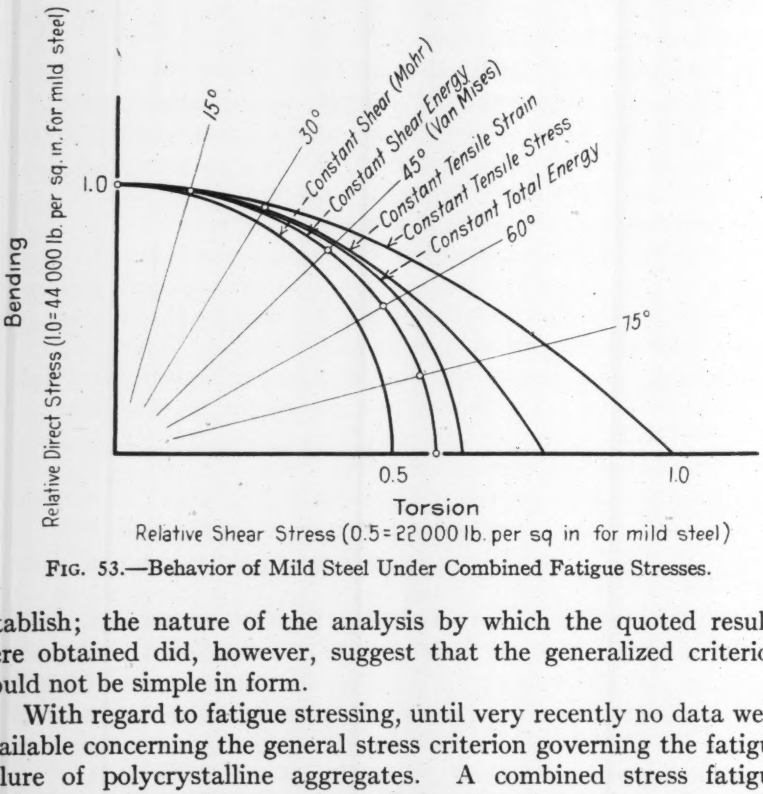

FIG. 53.—Behavior of Mild Steel Under Combined Fatigue Stresses.

With regard to fatigue stressing, until very recently no data were available concerning the general stress criterion governing the fatigue failure of polycrystalline aggregates. A combined stress fatigue testing machine(80) has now been designed and constructed, at the National Physical Laboratory, which enables the whole range of combined alternating bending and alternating torsion (cycles of reversed stresses) to be explored. Results obtained using a low-carbon steel (Fig. 53) show that the fatigue failure of this material conforms very closely indeed to the von Mises relation. The significance of this result with respect to the fatigue characteristics of the single crystal must be deferred until more evidence is obtained of the influence of the intercrystalline boundaries, also a variety of materials will be tested before any general conclusion can be drawn. In the meantime, it is of interest to observe an additional indication of the close connection that exists between plastic deformation and fatigue failure.

In conclusion, I would express the hope that, however inadequate has been the treatment of the subject, I have been successful in conveying an impression that the causes of failure of metals, which are of such importance to all branches of industry, are intimately associated with the crystalline structure of these metals and can be profitably studied from that aspect.

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# SYMPOSIUM ON CAST IRON

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HELD AT A JOINT MEETING OF THE
AMERICAN FOUNDRYMEN'S ASSOCIATION
AND THE
AMERICAN SOCIETY FOR TESTING MATERIALS
JUNE 26, 1933

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## PREFACE

The American Foundrymen's Association and the American Society for Testing Materials in 1930 outlined a program for the purpose of providing the engineering profession with authoritative data and information concerning the properties of metal castings. The program called for Symposiums on the various groups of cast metals. Malleable iron castings were covered in 1931, steel castings in 1932, with the present symposium being devoted to cast iron.

The Advisory Committee, appointed by the two societies to supervise this Symposium, had the following personnel:

D. M. Avey, *The Foundry*.
A. L. Boegehold, General Motors Corporation Research Laboratory.
J. W. Bolton, The Lunkenheimer Co.
H. Bornstein, *Chairman*, Deere and Co.
F. B. Coyle, International Nickel Co.
H. W. Gillett, Battelle Memorial Institute.
F. P. Gilligan, Henry Souther Engineering Co.
A. E. Hageboeck, Frank Foundries Corp.
R. E. Kennedy, *Secretary*, American Foundrymen's Assn.
J. T. MacKenzie, American Cast Iron Pipe Co.
R. S. MacPherran, Allis Chalmers Manufacturing Co.
O. Smalley, Gray Iron Institute.
E. K. Smith, Electro Metallurgical Co.
E. R. Young, Climax Molybdenum Co.

The chairman of the Advisory Committee appointed an Editorial Committee to review and revise the initially submitted material and then to submit the report to the Advisory Committee for approval.

The Editorial Committee was composed of the following personnel:

H. Bornstein, *Chairman*
R. E. Kennedy, *Secretary*
J. W. Bolton
J. T. MacKenzie
R. S. MacPherran
E. K. Smith

In the preparation of the initial material the helpful assistance of the following is acknowledged:

D. M. Avey, *The Foundry.*
A. L. Boegehold, General Motors Corporation Research Laboratory.
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E. F. Cone, *The Iron Age.*
F. B. Coyle, International Nickel Co.
J. Erler, Farrel-Birmingham Co.
H. W. Gillett, Battelle Memorial Institute.
F. P. Gilligan, Henry Souther Engineering Co.
A. E. Hageboeck, Frank Foundries Corp.
Rebecca Hall, Lakeside Malleable Iron Co.
W. W. Kerlin, Gray Iron Institute.
M. Kuniansky, Lynchburg Foundry Co.
J. T. MacKenzie, American Cast Iron Pipe Co.
R. S. MacPherran, Allis Chalmers Manufacturing Co.
G. Phillips, Frank Foundries Corp.
W. H. Rother, Buffalo Foundry and Machine Co.
O. Smalley, Gray Iron Institute.
E. K. Smith, Electro Metallurgical Co.
W. H. Spencer, American Cast Iron Pipe Co.
Jerome Strauss, Vanadium Corporation of America.
E. R. Young, Climax Molybdenum Co.

Due to the necessity for keeping the amount of material to a minimum, the original material as submitted has had to be considerably condensed, but it is hoped that the objective of presenting a concise authoritative review of cast iron, its properties and applications has been accomplished.

H. BORNSTEIN,
*Chairman, Advisory Committee*
*on Symposium.*

# SYMPOSIUM ON CAST IRON

## I. INTRODUCTION

JUNE 26, 1933.

Cast iron, the first of the ferrous metals to be cast, has for some two centuries had a place of great importance in the industrial world. It was not, however, until well after the close of the nineteenth century that cast iron founding became more of a science than an art. Competition with more recently developed materials has been of inestimable worth in placing cast iron production on a scientific basis, thus broadening the range of working properties, instituting accurate technical control and resulting in economical production methods.

Today, cast iron is a reliable engineering material with great possibilities for further development in the near future.

### DEFINITIONS

A satisfactory definition of "cast iron," as with so many metallurgical products, is not available. To the average layman the term signifies a hard, brittle material. However, cast iron has been developed to such a point that the general term now covers a wide range of iron-carbon-silicon alloys containing from 2 to 4 per cent carbon and 0.25 per cent or more silicon in combination with varying percentages of manganese, sulfur and phosphorus, and sometimes one or more of the special alloying elements such as nickel, chromium, molybdenum, vanadium and titanium.

*Cast Iron.*—In general, cast iron has been defined as an iron containing so much carbon or its equivalent that it is not malleable as cast.

Cast irons may be grouped broadly into three classes: (1) Gray iron, (2) white iron, and (3) chilled iron.

*Gray Cast Irons* are irons having a composition in which a large portion of the carbon is distributed throughout the casting as free or graphitic carbon in the flake form.

*White Irons* are irons of such composition that, when cast and broken, the resulting fracture is white in appearance, due to the fact that all the carbon is in the chemically combined form of iron carbide ($Fe_3C$). In this form the iron is extremely hard and brittle. Most white iron castings are produced in malleable foundries, where they are so heat treated as to become malleable cast iron. Some are used in white iron form for such products as plow shares, grinding plates and grinding balls, where resistance to wear is of the greatest importance.

*Chilled Cast Irons* are irons with some section purposely cooled so fast by means of chills that the carbon is retained in the combined form or as white iron, and with the other sections cooled naturally and having the carbon in the form found in gray cast iron. Between the white chilled portion and the gray portion lies a region of so-called mottled iron, in which some areas are white iron and some gray iron. A chilled roll is an example of this type of iron.

*Semi-Steel* is a term originally used to designate a cast iron made from a mixture of iron and some steel scrap. While this term is still quite common, it should not be used as it is misleading, the product having none of the characteristics of steel and being strictly a cast iron. Even when melting all steel charges in the cupola, as is sometimes done, the absorption by the steel of carbon from the coke charges will produce a cast iron.

## HISTORICAL

Although cast iron was the first ferrous metal to be cast, no definite information is available as to when this occurred. One theory is that the early furnace men, when making wrought iron, unquestionably must have noticed occasional molten metal which they considered a serious detriment. Only when they found that this metal could be successfully cast into cannon balls (about 1388 A. D.) was a new industry begun.

Actually, cast iron was known in Asia much earlier, as there are records of a bridge with cast iron chains built in Japan in 70 A. D. The first recorded example of pig iron making goes back to 1311 A. D. in the Siegerland of Westphalia.

The art of making iron castings first appeared in England before 1500 A. D., the first records being of casting guns. German records show that cannons were cast of white iron as early as 1414.

Soon after the use of iron for cannon became prevalent, plates were cast for open fireplaces. Many of these, known as "fire-backs," still are in existence in this country and in Europe. One of the earliest cast stoves was made in a German monastery in 1548. Andirons also made their appearance in cast metal about this time.

From the time that commercial smelting methods first were employed, foundry work was for a long time closely linked with blast-furnace operations. In fact, the only ferrous castings made in the seventeenth century and until late in the eighteenth century were produced from direct metal at the blast furnace. Fire-backs, stove plates, hollow ware, cannon balls, and many other cast-iron products were cast in the sand beds in front of blast furnaces.

The first cupola for remelting pig iron and scrap has been attributed to the Frenchman Reamur before 1750. Ease of operation and economy in melting soon made the cupola process popular in Europe.

In America, castings production began with colonization. Probably the first commercial casting made in this country, a cooking pot of colonial design, now is the property of the city of Lynn, Mass. It was made at the Saugus Iron Works in 1642. Gradually the foundry industry spread through New York and Pennsylvania. In 1789 from 10 to 25 tons of iron castings were being produced weekly by 14 furnaces, many of which cast cannon and round shot during the American Revolution.

The first cupola was introduced in this country in Pennsylvania in 1820. Thereafter the spread of foundries was rapid, since they no longer had to be operated in conjunction with blast furnaces.

With the development of our mechanical industries, especially in the railroad and machine-tool fields, casting practice kept pace and played an important part in providing the necessary metal parts. For many years, however, foundry practice remained largely an art rather than a science, and the foundry product was not greatly improved until the latter part of the nineteenth century. Not until then was chemical control found possible and molding machines introduced to meet the demand for quantity production of castings from the same patterns.

Demands for the quantity production of stove and agricultural machinery castings followed by demands of the automotive industry probably were as influential as any other factors in changing iron founding from an art into a science. Rapid development of testing methods, and demands for properties in excess of those found in the earlier cast irons, brought about greater technical control and metallurgical knowledge in the foundry, enabling it to keep pace with exacting engineering requirements.

In addition, the use of alloys and developments made in heat treating and furnace practice during the past few years have added greatly to the possibilities of securing increased properties and uniform quality in cast iron. Ten years ago cast irons with tensile strengths of 40,000 lb. per sq. in. were very unusual. Today strengths of 45,000 to 55,000 lb. per sq. in. are being produced regularly in many foundries. Cupola iron of 70,000 lb. per sq. in. and special irons of even higher strengths are being made by some firms in air, electric and rotary furnaces.

Perhaps no more striking illustration of the change in the traditional attitude toward cast iron can be cited than that expressed in a recent editorial of a technical journal, when it stated: "Adoption by a leading automobile manufacturer of a camshaft cast from electric furnace iron, and the imminent use of a cast crankshaft and camshaft by another manufacturer, focus the spotlight on the swift developments recently made in high-strength alloy cast iron. It is significant and of great promise that this material is encompassing ever-widening vistas, with a growth little short of phenomenal in the motor-car industry, where production costs and long service life are major considerations. . . With a tensile strength as high as 70,000 lb. per sq. in. and a Brinell hardness of 300, it is displacing forged steel in parts where a short time ago metallurgists would have pronounced it impracticable."

## MANUFACTURE

The manufacture of cast iron involves these few essential steps: (1) Design, (2) preparation of pattern and core equipment, (3) molding, (4) core making, (5) melting and pouring, and (6) finishing and inspection.

### *Design:*

In designing parts for castings the engineer must consider such factors as internal shrinkage, sections giving greatest strength, gating, removal of core gases and supporting of cores, and draft to permit easy removal from the mold. These factors are considered in more detail in section VIII on Foundry Factors of Importance in the Production of Iron Castings.[1]

### *Pattern Equipment Production:*

Planning and making the proper pattern and core box equipment have an important bearing on securing good castings, especially when the minimum cost is considered. If the purchaser is to furnish the patterns, the foundry staff should be consulted as to the type and details of equipment to be made.

### *Molding:*

Molding consists of making the form into which the metal is to be cast. The most common type of molding is sand molding, the material used being sand in its "green" or moist state, and in its "dry" state. Loam molding is used in certain cases, the mold form being built up of a backing of brick coated with a special loam-sand mixture. The loam is applied in a wet condition, the mold being dried before final assembly. Other mold forms are permanent molds or "long life" molds. Centrifugal pipe molding made in all-metal molds is one type of permanent molding.

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[1] See p. 258.

*Green-Sand Mold.*—Where the mold material is a moist refractory sand and the metal is poured into the moist mold, the process is known as green-sand molding. This method is used in producing the bulk of iron castings.

*Dry-Sand Mold.*—Where the mold material, when shaped, is a moist refractory sand subsequently dried before the casting is poured, the process is known as dry-sand molding. This method is used largely in production of large castings.

The sand casting method is applicable to the production of any number of molds from a single pattern. Sand molds are made by hand or machine process, the method used depending on shape and size of the casting, number of castings to be produced, and facilities of the shop. Economies in production can be secured by consultation among the foundry, pattern, and design departments before the pattern is made.

*Permanent Molds.*—Permanent molds are made of metal or refractory material, the same mold being used over and over. Use of this type of mold is limited to fairly simple forms and to production in larger quantities. When this mold is of metal, castings generally are finer in grain than those made by the sand casting method because of more rapid cooling of the metal when poured.

*Centrifugal Molds.*—Centrifugal casting is a special form of permanent mold casting. Some of these molds are entirely of metal, others are lined with a refractory material. The molds are rotated rapidly while the metal is poured, forcing the metal to the inner mold surface by centrifugal force. Great tonnages of cast-iron pipe are made by this process.

### *Cores:*

The interior shapes of castings generally are formed by means of sand cores. Some cores are formed by the pattern itself, the core being made of green molding sand. Other cores of special core-sand mixtures are made in special boxes and then baked and inserted after the pattern has been withdrawn. Metal shapes also are sometimes used. In centrifugal casting, the main interior shapes are formed by metal flowing to the outside of the casting form, the interior being left as a cylindrical hole.

To simplify molding operations, baked cores sometimes are used to form exterior surfaces of castings.

*Melting:*

Cast iron melting usually is performed in one or another of the following types of furnaces, or by a combination of several types.

*Cupola.*—The oldest form of furnace for melting cast iron, and the cheapest melting unit, is the cupola. By using cupolas of different size and operating for long or short periods, any desired amount of iron can be obtained. The quality of the metal produced can be closely regulated by experienced operators, although quality is not so easily controlled in the cupola as in other types of furnaces where tests can be made of the molten metal in the bath and slags and suitable corrections made to refine the bath metal.

In the cupola, which is simply a vertical steel cylinder lined with a refractory material, alternate charges of coke and iron are placed, air forced through them and the iron melted by contact with hot gases; the iron drips down through the incandescent fuel to the hearth at the bottom of the furnace. In contact with the gases, the iron loses some of its manganese and silicon and picks up some sulfur. The carbon change depends on melting conditions and the composition of the charge. After the iron is placed inside the cupola, the operator has little further control over the composition of the metal that comes out at the spout except as may be secured by blast manipulation.

*Air Furnace.*—The air furnace has been used for many years in producing certain high-grade cast irons. The entire charge is placed in the furnace at one time, melted down in one mass on a hearth, and tapped out in a short space of time. During melting the metal is more subject to control than in the cupola process, since tests can be made of the melted metal on the hearth. Melting cost, however, is greater in the air furnace than in the cupola, and continuous pouring is not attainable except by duplexing.

*Electric Furnace.*—Because quality can be better controlled, higher pouring temperatures secured and cheaper materials used in the electric furnace, this type of equipment is being used to an increasing extent in melting cast iron. However, its melting cost is normally higher than that of cupola melting, so that it is generally used only when special high-quality iron is desired. In a few cases the cupola, because of its low melting cost, is used for melting, the metal then being transferred to the electric furnace for refining and raising the temperature for pouring control; this combination method is called "duplexing."

*Rotary Pulverized-Fuel-Fired Furnace.*—With the development of equipment for pulverizing fuel, a rotary furnace using pulverized coal was recently introduced in Germany. This type of furnace, with which the close control of composition essential to the production of high-strength irons is obtainable, and having a fuel cost approaching that of the cupola, has been used extensively in Europe and now is being tried out in this country also.

*Casting:*

The metal, when drawn from the furnace, is transferred in ladles to the molds where casting (pouring) takes place. The rate of pouring, determined by the size of the gate opening, depends on the size of the casting and its metal section. After the mold is poured and the metal set, the castings are removed from the molds and taken to the cleaning room.

*Cleaning, Finishing and Inspection:*

In cleaning the castings, adhering sand is removed by brushing, or tumbling in a revolving barrel, or by abrasive or water blasting. In a few cases, castings are pickled in some form of acid which removes the adhering sandy scale or oxide. When the casting is cleaned, the gates, risers and fins not already broken off in handling are removed and the rough places on the casting smoothed by means of chipping hammers or grinding.

Inspection takes place at several points in the cleaning process, as the castings are under almost continual observation. After being finished, the castings are given a final inspection.

*Heat Treating:*

Heat treating iron castings, when desired, is performed after the castings have been cleaned. The purposes and practices connected with heat treating are discussed fully in section V on Heat Treatment.[1]

## FIELD OF APPLICATION

Iron castings are used in practically every walk of life, as they can be produced cheaply, with good surfaces, true to pattern, and with surface details of firm, sharp outline. However, certain industries use the bulk of iron castings, and some of the more general fields of application are listed below in order to give some idea of the extent and variety of casting uses:

*Automotive.*—Cylinder blocks and heads, crankcases, flywheels, pistons, piston rings, camshafts, brake drums, and miscellaneous parts.

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[1] See p. 240.

*Pipe.*—Cast-iron pipe and fittings.

*Machine Tools.*—Beds and bases, and many other parts.

*Railroads.*—Chilled car wheels, brake shoes, cylinders, pistons, smoke stacks, etc.

*Hardware.*

*Architectural.*—Stair treads, railings, ornamental work, sash weights, etc.

*Heating.*—Radiators, fire pots, grate bars, stove parts, etc.

*Sanitary Ware.*—Bathtubs, wash basins, bowls, etc.

*Electrical.*—Dynamo frames, flywheels, base frames, etc.

*Hammer and Forming Dies.*

*Power Plants.*—Flywheels, engine bases, cylinders, furnace frames, firebox parts, grate bars, etc.

*Agricultural.*—Wheels, gears, sprockets, plowshares, plow points, pulleys, frames, etc.

*Rolling Mills.*—Rolls, ingot molds, slag ladles, etc.

*Marine.*—Engines, pillow blocks, etc.

*Printing.*—Press frames, rolls and parts, etc.

*Municipal.*—Manhole covers, fireplugs, lamp posts and bases, pipe and fittings, tunnel segments, catch basins, mail boxes, traffic signs, etc.

*Toys.*

*Ornamental.*—Andirons, fire sets, lamp bases, book ends, garden benches, road signs, calendar bases.

*Chemical.*—Retorts, acid pans, sulfanators, acid eggs, concentrating pans, evaporators, etc.

*Mining.*—Sludge pumps, grinding and crushing equipment, wear plates, chutes, etc.

*Petroleum.*—Valves, pipe, fittings, etc.

With the recent advent of alloys and special mixtures, giving unusual properties in cast irons, many new combinations constantly are being discovered.

## SCOPE OF SYMPOSIUM

In the following sections of this work an endeavor has been made to present concise authoritative data on the composition, metallurgy and properties of the many grades of cast iron which are now available. Cast iron being a complex material, the section on Metallurgy has been prepared to show the effects of the various elements which are normally in cast iron and of the alloy additions which are playing such an important part in adding to or increasing the available properties of cast iron.

The section on Classification and Specifications[1] treats only of those irons which have been brought under the A.S.T.M. Specifications. Many special classes of irons of more recent development are available and some of these are mentioned in other sections. The properties of cast iron of interest to the engineer are covered first for general or normal cast irons and secondly as they are found in the special cast irons for corrosion and heat resistance and for wear and machineability.

Heat Treatment,[2] White and Chilled Irons,[3] are dealt with in separate sections. In addition, two sections, those on Foundry Factors of Importance in the Production of Iron Castings,[4] and Welding,[5] have been added as these subjects seem logical for inclusion in such a general treatise on cast iron.

## II. METALLURGY OF CAST IRON

The term cast iron is applied to a series of alloys of iron, silicon and carbon, which also contain appreciable percentages of other elements such as manganese, phosphorus and sulfur. Elements such as nickel, chromium, molybdenum, copper, titanium, etc., usually termed alloys, are not infrequently added with the intention of modifying certain properties of the metal.

Most commercial cast irons contain between 2.50 and 3.75 per cent of total carbon and silicon from about 0.25 per cent up to 3.00 per cent. White, chilled, and malleable cast irons usually contain less than 1.00 per cent of silicon. Some very heavy gray-iron castings occasionally are made with silicon content below 1.00 per cent, but the majority of gray-iron castings contain silicon above 1.00 per cent and below 2.75 per cent.

### NORMAL ELEMENTS

*Forms of Carbon:*

Carbon in cast irons is found in three or more forms. In fully white irons and the chilled portions of chill cast irons, most of the carbon occurs as free iron carbide, or massive cementite as it is called. Cementite is a carbide of iron, its formula being $Fe_3C$. It is very hard and rather brittle, which characteristics are in large part transmitted to those irons in which free cementite is a major structural component. In very soft gray irons most of the carbon occurs as free graphitic carbon, a weak, soft form of inclusion. The matrix of the majority of commercial gray irons consists of pearlite, an eutectoid of ferrite (structurally pure iron) and iron carbide. This component pearlite possesses considerable strength, wear resistance and other desirable properties.

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[1] See p. 238. [2] See p. 240. [3] See p. 247. [4] See p. 258. [5] See p. 252.

*Factors in Control of Properties:*

The metallurgist controls the properties of his cast iron through control of the amount, size and distribution of the various carbon formations. This he accomplishes through control of composition (particularly of carbon and silicon) and other factors. The factors influencing the properties of cast iron are:

1. Chemical composition—initial, incidental and final.
2. Structural and mechanical make-up of charge.
3. Melting process.
4. Thermal and mechanical history—furnace spout to cooled casting.
5. Subsequent heat treatment.
6. Design and workmanship.

With factors 2, 3, 4, 5 and 6 constant, the properties of the metal depend upon its composition. Where a foundry makes its own castings, factors other than composition are controlled and for most practical purposes may be considered constant. Work specifications therefore may quite logically include composition. When castings are purchased in the open market, and various foundries are quoting, an initial chemical specification usually proves of little practical value. *Different foundries, employing different methods and different compositions, may produce irons of substantially the same engineering properties. In such cases extensive chemical specifications frequently are unduly restrictive. For this reason A.S.T.M. Specifications A 48 – 32 T*[1] *do not recognize chemical specifications.* Once a satisfactory source of castings has been found, a supplementary chemical specification may be useful as a check on the uniformity of product.

The engineer is interested in the mechanical properties of the metal, and, as long as these are satisfactory, the means used to attain them are of secondary importance.

*Influence of Carbon:*

The engineering properties of any class of cast iron depend upon its structure and its soundness. The structure of a given iron depends very largely upon the amount, size and distribution of the various forms of carbon. The relationship of these structural formations to engineering properties is covered more completely under Structural Components.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 625 (1932); also 1932 Book of A.S.T.M. Tentative Standards, p. 179.

The total carbon content in a given iron depends on the carbon in the metal charge and on the loss or absorption, as the case may be, of carbon occurring during melting. In the manufacture of pig iron the molten iron readily absorbs or dissolves carbon from the coke, and in most cases is fully saturated with carbon by the time it is tapped from the hearth. The amount of carbon so dissolved depends principally on the temperature and on the amount of silicon and phosphorus present in the metal.

The amount of carbon which can be dissolved by molten iron increases with increase in temperature(**1**).[2] At the freezing point the amount of carbon soluble in pure iron usually corresponds to the eutectic percentage, 4.3 per cent carbon. Silicon lowers the eutectic about 0.30 per cent carbon for each 1 per cent of silicon present. Therefore, the higher silicon pig irons usually are lower in total carbon content. Phosphorus also tends to lower the total carbon content in the amount of 0.30 per cent carbon for each 1 per cent of phosphorus.

Foundry returns and purchased scrap normally contain less total carbon than do pig irons. The carbon percentages in foundry returns of course are or should be known. Ordinary purchased scrap may run anywhere from 3.00 to 3.75 per cent total carbon. Steel scrap usually runs 0.10 to 1.00 per cent carbon, soft steel being in the lower range and hard steel in the higher.

During the remelting processes used in production of castings, the carbon content is changed, and a certain degree of equilibrium is attained. Some of the oxidizing gases resulting from the air used in the cupola and air-furnace processes tend to reduce the carbon content of the charged metals whereas reducing gases and incandescent coke may cause carbon pickup. The adjustment of various factors to insure reasonably constant carbon content demands experience and close supervision.

From an engineering viewpoint irons of lower carbon content (2.75 to 3.25 per cent) usually are preferred to irons of high carbon content (about 3.25 up to 3.75 per cent) since the lower carbon metals usually are stronger and vary less in properties over a range of section. Lower carbon irons usually exhibit lower fluidity (at the same pouring temperature) and more careful melting and molding practice is required to offset dangers from contraction and shrinkage. However, lower carbon irons can be and are made quite sound and free from internal defects. Because of their greater strength and hardness, lower carbon irons usually are somewhat more difficult to machine. However, the modern trend toward use of lower carbon - higher silicon irons permits better machineability, for given strength, than usually is attained with higher carbon - lower silicon alloys.

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[1] See p. 133.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended hereto, see p. 268.

The nature of the various carbon formations and the influence of total carbon content is discussed in greater detail under Structural Components.[1]

*Influence of Silicon:*

Silicon tends to break down the combined or cementitic carbons in cast irons and is the predominant element in determining the relative proportions of combined and graphitic carbons. Other factors equal, the higher the silicon content the greater the relative amount of graphite and consequently the less combined carbon. Thus with given operating conditions and approximately the same total carbon content, the foundryman controls the properties of his metal largely by means of silicon adjustment. Thus if a softer lower-strength iron is desired, the silicon is increased, and *vice versa*. If, in an attempt to get maximum strength, the silicon is decreased too much, chilled spots and consequent unsatisfactory machineability are a likely result. This applies up to a silicon content of about 3.0 per cent. As mentioned above, increase in silicon content lowers the solvent power of iron for carbon.

According to the latest evidence, silicon dissolves in the iron forming a matrix which is brittle and rather hard. The presence of this dissolved silicon in the matrix up to a certain point adds to its strength and hardness. However, somewhat beyond 3 per cent silicon it completely displaces the iron carbide and we have a material consisting of graphite in a matrix so high in dissolved silicon that it is hard, weak and brittle. For example, at about 4.0 per cent silicon the iron has a glazed fracture, carbon content is reduced to about 3.08 per cent and the metal is comparatively worthless for engineering purposes, except for heat resistance.

A comparatively few years ago the influences of total carbon on cast iron were not well understood by most foundrymen, and carbon control methods were neglected. Silicon was the controlling factor to many foundry mixture makers. Today through increased knowledge of the effects of carbon content and other factors, silicon contents, while carefully regulated, may vary greatly from those formerly specified. For example, most modern high-test irons are low in total carbon, and in these irons much higher silicon contents are permissible, or in fact are needed, than is proper for higher carbon irons.

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[1] See p. 133.

The known effects of silicon may be summarized as follows:

1. It decreases the stability of iron carbide (cementite) thus promoting graphitization.

2. It lowers the percentage of carbon required for the eutectic. This materially alters the "solubility" of carbon under given conditions of temperature, etc., and has an influence on the general distribution of the structural components.

3. The deoxidizing action of silicon probably has an indirect effect on graphitization and also may affect the fluidity of cast iron.

4. It changes the temperature of both eutectic and eutectoid transformations, but this effect may be masked by the presence of other elements, notably manganese.

5. It lowers the percentage of carbon required for the eutectoid pearlite.

6. In excess it produces a weak brittle matrix and practically complete graphitization.

### *Influence of Manganese:*

The manganese content of most commercial cast irons is between 0.40 and 1.00 per cent. Broadly speaking, within this range variations in manganese content apparently do not have very marked effect on the properties of the alloy if silicon content is ample. Irons above 1 per cent manganese furnish an interesting field for the foundry metallurgist, and all the effects of higher manganese content (say 1.00 to 2.50 per cent) have not been completely determined for a wide variety of American irons.

The first action of manganese in cast iron is combination with the bulk of sulfur present. Manganese and sulfur form the compound, manganese sulfide, which is relatively insoluble in molten and in solid iron. Manganese sulfide, plus other substances, for example, manganese silicates, etc., is found as dove or blue-gray colored inclusions. These inclusions are lower in specific gravity than the molten metal. Therefore, they tend to float to the top of the liquid—a fact which explains the possibility of partial desulfurization of iron standing in a ladle, and the usually higher sulfur and manganese content on the cope side of heavy castings. For "complete" combinations of sulfur, an excess of several tenths of a per cent manganese above the amount theoretically necessary is required. Thus manganese in large degree inhibits the effects of sulfur. (See section on Influence of Sulfur below for comments on effects of sulfur.)

Another type of inclusion, presumably manganese silicate, may occur in considerable amount when silicon and manganese contents are approximately equal. This type of inclusion causes glazed spots and porous areas.

The balance of the manganese (that is, manganese not in inclusion form) generally is considered as occurring as the carbide $Mn_3C$, in irons containing combined carbon. This carbide is intimately associated with the iron carbide ($Fe_3C$). In irons of ferritic matrix the manganese occurs in solid solution in the ferrite.

Manganese in itself tends to increase the stability of the carbide, although this effect is not appreciable in irons of the usual range of manganese content.

An increase in manganese content lowers the solidus somewhat, and also lowers the pearlitic transformation point. It also is claimed that it slightly reduces the combined carbon required for a pearlitic matrix. In case of lower carbon - lower silicon alloys, increase of manganese to well above 10 per cent causes the metal to become austenitic. Therefore, we would logically expect that as the manganese content of cast iron is increased the matrix is successively pearlitic, sorbitic, martensitic, and austenitic. In a pearlitic iron increase of manganese from say 0.40 to 0.70 per cent would affect the structure of the matrix very little. With increase up to say 1.0 per cent, we would expect the matrix to appear somewhat sorbitic as is the case with the low alloy manganese steel of corresponding manganese content.

As a deoxidizer and scavenger, manganese in the usual ranges apparently exerts a beneficial effect, although the relatively high carbon and silicon content of cast irons suggests that these *per se* may furnish the requisite degree of deoxidization.

Increase in manganese increases the total contraction and shrinkage slightly. Tensile strength, other factors equal, apparently reaches a maximum at about 1.0 per cent.

### *Influence of Sulfur:*

Until the last few years the presence of an appreciable percentage of sulfur in iron castings was viewed with suspicion and material containing over 0.10 per cent was barred by a number of leading specifications. Without a doubt, the influence of this element was over-emphasized and its potentialities were even somewhat exaggerated. Better understanding of the effects of other elements, notably carbon, and the actual production of excellent high strength irons with sulfur content well over 0.10 per cent have dissipated much of this prejudice. The majority of commercial irons contain sufficient manganese to neutralize the effects of sulfur, as explained under manganese, if sufficient time for completion of the reaction is allowed. The resulting inclusion, manganese sulfide, while not beneficial, occurs in such small percentages by volume that its effect on physical properties is nearly, if not completely, masked by the predominating effect of the major inclusions in gray iron, notably graphite flakes. The sulfur present in cupola melted gray irons is derived from the metals charged and from the coke. In the air process used for production of malleable iron and some gray irons sulfur pickup from the fuel is possible. In malleable annealing there also may be some pickup.

Up to 0.15 per cent, sulfur is not harmful in the majority of gray-iron castings. When melting practice is poor, particularly when excess air is used, blowholes and dirty castings are a frequent occurrence. This condition is aggravated by high sulfur content. In this case sulfur may form volatile or gaseous oxides, or so-called oxy-sulfides. These and the oxides of carbon form blowholes and pinholes. In some special-purpose castings, sulfur content is a more important factor and may require more rigid control.

In the absence of sufficient manganese, sulfur increases the stability of the carbide very markedly, thus inhibiting graphitization. This action is lessened with increase in silicon content. Even in the presence of manganese, iron of high sulfur content, when cast against chills, shows a greater tendency to chill deeper than iron of lower sulfur content, other factors equal.

*Influence of Phosphorus:*

Phosphorus is present as an "impurity" in all cast irons and also may be introduced for the purpose of rendering the iron more fluid.

In presence of phosphorus the structural component commonly termed steadite is found in cast irons. The exact nature of this component varies somewhat. In chilled and mottled irons a considerable portion of this phosphorus-rich component consists of a ternary eutectic of iron, cementite and iron phosphide, while in many gray irons it consists largely of a binary eutectic formation.

In this symposium the term will be used to indicate all phosphorus-rich components.

Steadite is a hard, brittle component of rather low melting point (about 1750 to 1800 F.). It contains about 10 per cent phosphorus, and its specific gravity is not far different from that of the iron itself. Therefore, about 1 per cent of phosphorus indicates the presence of about 10 per cent steadite by volume. As might be expected the presence of considerable percentages of phosphorus modifies the properties of cast iron quite materially. The effects may be summarized as follows:

1. Phosphorus forms approximately ten times its percentage of steadite. This component, being of low melting range, adds materially to the fluidity of cast iron. Hence, high-phosphorus irons, other factors equal, are more fluid than low-phosphorus irons.

2. Due to the hardness of steadite, increase in phosphorus diminishes machineability. This effect is more apparent in higher strength irons.

3. Within the usual range of American practice (0.15 to 0.90 per cent phosphorus) this element has no very marked effect on static strength of most commercial irons. Where very high strength is required phosphorus must be kept low. Due to the tendency of steadite to form a network formation, high-phosphorus irons show lower ultimate deflection values than do lower-phosphorus materials. The intermediate deflections (a measure of rigidity) are little affected by changes in phosphorus content. The prevailing impression is that higher-phosphorus irons are more brittle than others of otherwise similar type. This impression is borne out by the impact test investigations(2) being conducted by the A.S.T.M.

4. Phosphorus lowers the amount of carbon required for the eutectic, the lowering amounting to 0.3 per cent carbon for each 1 per cent of phosphorus.

5. Increase in phosphorus content lowers the initial freezing point of cast iron, lowers the eutectic freezing point, increases the intensity of the third or steadite solidification point, and has little if any direct effect on the pearlitic transformation point.

6. Due to the fact that steadite occupies some volume in the matrix, it is a natural consequence that increase in phosphorus decreases the amount of combined carbon essential to production of an eutectoid or pearlitic matrix.

7. In general, phosphorus increases contraction slightly but this effect is negligible compared with the effects of graphitization.

8. Apparently phosphorus has little direct effect either in increasing or diminishing graphitization, except in some cases of very light castings.

## STRUCTURAL COMPONENTS

The microscope reveals that most commercial gray irons contain four major structural components, namely, *graphite, pearlite, ferrite and steadite.* White and mottled irons contain free cementite and pearlite (or one of its transition forms). Mottled iron also contains some graphite. Properly annealed malleable irons consist of graphite and ferrite. All cast irons contain minor inclusions, of which manganese sulfide is the most common. Since the engineering properties of a casting depend on the structural make-up of the material and the degree of soundness of the casting, study of structure is important. The proportions of structural components in some typical irons are given in Table I.

TABLE I.—PROPORTIONS OF STRUCTURAL COMPONENTS IN SOME TYPICAL IRONS.

| Description[a] | Chemical Composition, per cent | | | | | | |
|---|---|---|---|---|---|---|---|
| | Graphite | Combined Carbon | Silicon | Manganese | Phosphorus | Sulfur | Iron |
| Type No. 1 | 2.10 | 0.80 | 1.10 | 0.75 | 0.20 | 0.07 | 94.98 |
| Type No. 2 | 2.50 | 0.70 | 1.80 | 0.80 | 0.32 | 0.10 | 93.78 |
| Type No. 3 | 2.65 | 0.65 | 1.25 | 1.00 | 0.10 | 0.12 | 94.23 |
| Type No. 4 | 2.50 | 0.72 | 1.50 | 0.90 | 0.30 | 0.11 | 93.97 |
| Type No. 5 | 3.09 | 0.40 | 2.40 | 0.55 | 1.05 | 0.10 | 92.41 |
| Type No. 6 | .... | 3.30 | 0.60 | 0.52 | 0.50 | 0.15 | 94.93 |
| Type No. 7 | 1.50 | 1.80 | 0.92 | 0.36 | 0.22 | 0.13 | 95.07 |

| Description[a] | Structural Composition, per cent by Volume | | | | | | | Calculated Specific Gravity |
|---|---|---|---|---|---|---|---|---|
| | Silico-Ferrite | Pearlite | Manganese Sulfide | Steadite | Graphite | Iron Sulfide | Free Cementite | |
| Type No. 1 | 6.36 | 84.59 | 0.35 | 2.00 | 6.70 | .... | .... | 7.34 |
| Type No. 2 | 15.65 | 72.84 | 0.49 | 3.15 | 7.87 | .... | .... | 7.23 |
| Type No. 3 | 21.98 | 67.89 | 0.60 | 0.98 | 8.35 | .... | .... | 7.25 |
| Type No. 4 | 12.98 | 75.64 | 0.54 | 2.96 | 7.88 | .... | .... | 7.25 |
| Type No. 5 | 39.14 | 40.75 | 0.48 | 10.12 | 9.51 | .... | .... | 7.08 |
| Type No. 6 | 2.23 | 49.13 | 0.67 | 5.21 | none | 0.09 | 42.67 | 7.66 |
| Type No. 7 | 3.30 | 72.71 | 0.45 | 2.23 | 4.84 | 0.21 | 16.26 | 7.43 |

[a] Types Nos. 1 to 5, inclusive, are gray irons, while No. 6 is a white iron and No. 7 is a mottled iron.

*Nature of Structural Components:*

*Graphite.*—Structurally speaking, gray iron consists of a coherent metallic matrix throughout which numerous graphite flakes are dispersed. Since these graphite flakes possess little tenacity, and since they destroy much of the continuity of the matrix, their potentialities for affecting the mechanical properties of the metal are very

FIG. 1.—Representative Sample of Cast Iron Showing Coarse Graphite, Unetched (× 100). (Courtesy of Lunkenheimer Co.)

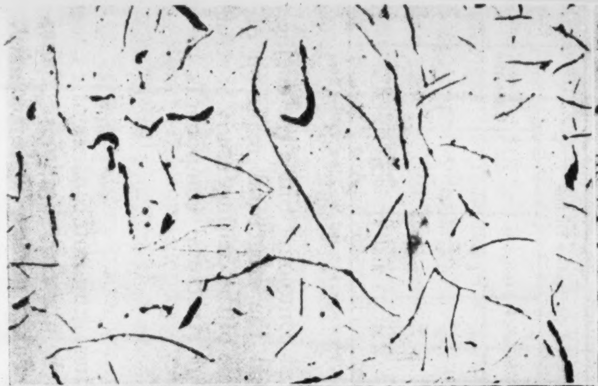
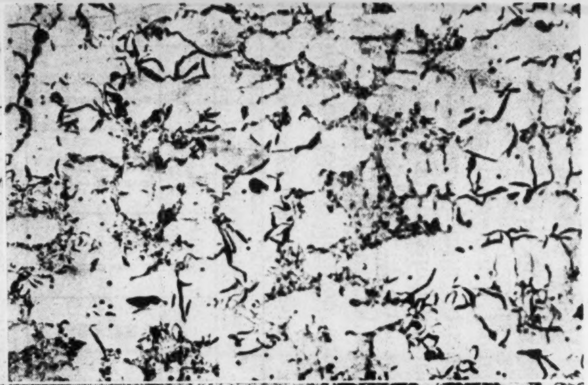


FIG. 2.—Representative Sample of Cast Iron Showing Fine Graphite, Etched (× 100). (Courtesy of Lunkenheimer Co.)



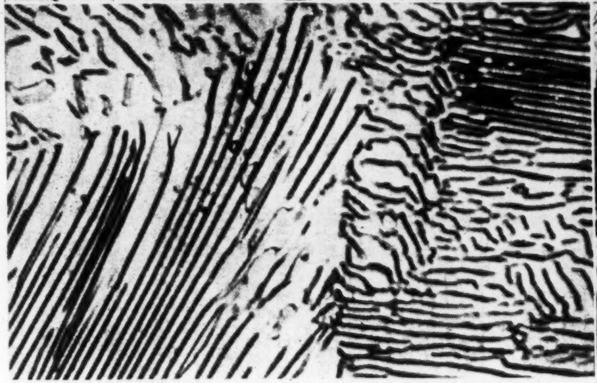


FIG. 3.—Structure of Cast Iron Showing Pearlite, Etched (× 2000). (Courtesy of Lunkenheimer Co.)

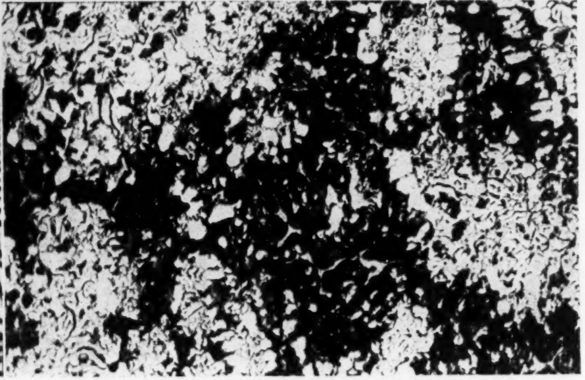


FIG. 4.—Cast Iron with Ferrite and Pseudo-Eutectic Graphite, Surrounded by Pearlite, Etched (× 100). (Courtesy of Lunkenheimer Co.)

**FIG. 5.**—Iron Cast Into Heavy Sections Showing Large Graphite Flakes Surrounded by Ferrite, Etched (× 100). (Courtesy of Electro Metallurgical Co.)

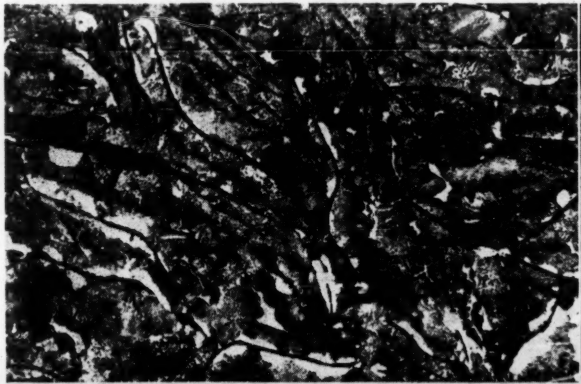
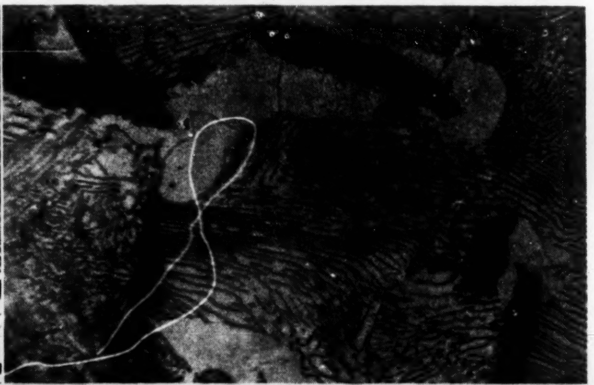


**FIG. 6.**—Cast Iron Structure Showing Ferrite Along Graphite Flakes with a Pearlitic Background, Etched (× 1000). (Courtesy of Lunkenheimer Co.)



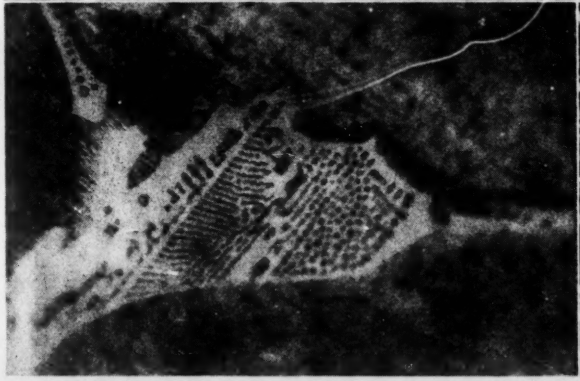


**FIG. 7.**—Cast Iron Showing Steadite, Etched (× 735). (Courtesy of Lunkenheimer Co.)

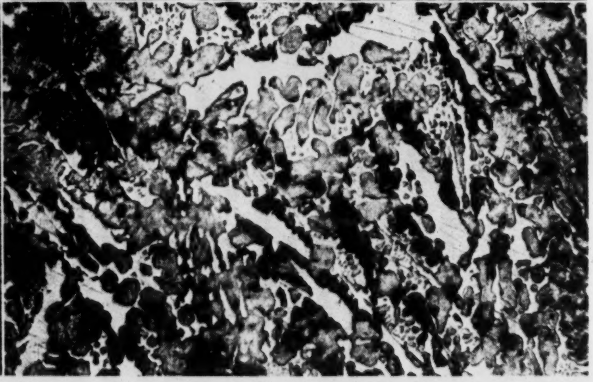


**FIG. 8.**—Structure of a Mottled Cast Iron. The white portions are cementite (× 100). (Courtesy of Lunkenheimer Co.)

great. The effects of graphite flakes depend on their amount, size, shape and distribution. The specific gravity of graphite flakes is low (about 2.25, *International Critical Tables*), hence they occupy more volume than would be indicated by ordinary gravimetric analysis. For example, an iron with 3.0 per cent graphite by weight would have about 9.6 per cent or more graphite by volume. The size of the individual flakes varies considerably, even in the same sample. The effectiveness of the flakes bears a relationship to the average flake size. Where variation is considerable it may be detected by the microscope (Fig. 1, coarse graphite flakes, and Fig. 2, more finely divided graphite flakes).

The various graphite flake shapes and groupings found in gray iron have been classified (3). Few irons are composed wholly of one graphite formation and the classifications are qualitative. However, the nature of the formation is directly related to the position of the alloy in the iron-carbon diagram.

The microscope shows the graphite flake formations in one plane only and to interpret the effects of the various groupings these must be considered from a three-dimensional viewpoint.

With other factors equal, increase in amount of graphite, increase in flake size, and unfavorable distribution, each affect the strength of the metal adversely. Where cooling rate is slow, highly graphitic irons frequently are so open grained that they appear pitted on machined surfaces. Such metal does not wear well unless unit pressure is low. With other factors equal, irons with lower graphite percentage and uniform distribution of rather small flakes are higher in tensile strength, have higher Brinell hardness numbers and are denser. To get best metal, all factors (amount, size and distribution) must be favorable. Many examples show that irons with small or fine individual flake size but with unfavorable distribution may be and often are inferior to metal with somewhat larger individual flake size yet better flake distribution.

*Pearlite.*—In normal gray irons the combined carbon occurs in the matrix formation known as pearlite (Fig. 3). This pearlite (composed of alternate lamellæ of ferrite and cementite) is quite similar in many respects to the pearlite found in carbon steels. Due to the presence of certain structural formations such as graphite and steadite and to the presence of considerable silicon and manganese a very largely pearlitic matrix in gray irons may be found with combined carbon anywhere from 0.50 to 0.90 per cent. (The "pure" eutectoid pearlite of the iron-carbon series contains about 0.85 per cent combined carbon.) There has been much propaganda relative to the value of a pearlitic matrix in gray irons. A pearlitic matrix is a very desirable characteristic in high-quality irons, yet a pearlitic matrix is no guarantee of high strength or other desirable properties. The pearlitic condition in cast iron is a relatively stable one and persists over a wide range of composition and section thicknesses.

Pearlite is strong (tensile strength about 120,000 lb. per sq. in.) and fairly hard (200 to 225 Brinell hardness). Most high-strength irons contain a pearlitic, or even a sorbitic matrix, but a pearlitic matrix is not, in itself, a guarantee of maximum physical properties. In some cases the amount and condition of the graphite are predominant factors. The unique wear-resisting properties of gray irons are well known to most engineers. Many tests have shown that for most conditions best wear resistance is obtained with higher-strength irons of pearlitic matrix.

*Ferrite.*—Irons of rather high silicon content or which have been cooled slowly may not have sufficient combined carbon to have an effectively pearlitic matrix. Under such circumstances free ferrite may occur in the matrix. This ferrite is found along the graphite flakes. The two examples (Figs. 4 and 5) show respectively an iron of rather high silicon content with ferrite and fine graphite formation intimately associated and an iron cast into a heavy section (hence cooled slowly) showing large graphite flakes surrounded by ferrite. The structural component ferrite is itself soft, ductile and only moderately strong (tensile strength about 50,000 lb. per sq. in. and 40 per cent elongation). In gray irons the continuity of ferrite is so broken up by graphite flakes that highly ferritic gray irons are not truly ductile, although they usually have higher deflections than irons of more fully pearlitic matrix, other factors approximately equal. The total carbon in gray irons occurs in two forms, combined or pearlitic, and free or graphitic. It then is obvious that with a given carbon content, the amounts of pearlite and graphite vary inversely, that is, the more graphite, the less pearlite and *vice versa.* The presence of ferrite means less pearlite and more graphite. Hence, ferritic irons, having less of the strong constituent pearlite, and more of the weakening constituent graphite, are not so strong as irons of pearlitic matrix, if total carbon content and section size are the same.

The general structure of a gray iron showing graphite, ferrite and pearlite is shown in Fig. 6.

*Steadite.*—The general nature of the phosphorus-rich component, steadite, was referred to in the section on phosphorus. The steadite in gray iron usually is composed largely of a binary cellular eutectic of iron and iron phosphide (Fig. 7) plus, in many cases, an excess structureless portion, consisting of solid solutions of iron and iron phosphide. It also was stated that in chilled and mottled irons, the phosphorus-rich component, when rejected is composed largely of a ternary eutectic of iron, iron phosphide and cementite. During the graphitization of gray iron the cementite of the ternary eutectic is rejected, leaving the binary formation. Recent researches show that the completeness of the change from the ternary to the binary form takes appreciable time. In very light castings of truly gray iron, cases occasionally arise when the cementite in the phosphorus formation is partially retained, although no free or massive cementite can be found. In the absence of combined carbon, phosphorus up to 1.7 per cent or thereabouts is soluble in ferrite. Therefore, if gray irons containing steadite are annealed at temperatures approaching 1800 F., the phosphorus dissolves in the ferrite and is not detected by routine microscopic methods. (This solution occurs at 1600 F. or even lower if annealing time is prolonged sufficiently.) Incidentally this phenomenon explains why steadite is not found in malleable irons, although found in gray irons containing only a few hundredths per cent of phosphorus.

If there is sufficient phosphorus present, under certain cooling conditions steadite forms a sort of network through the metal. Some have claimed that such a network formation is essential to high strength in high-phosphorus irons.

*Cementite and Austenite.*—Cementite is iron carbide and possesses the chemical formula $Fe_3C$. It contains 6.67 per cent carbon. Therefore, an iron containing 6.67 per cent carbon (and drastically chilled to prevent decomposition of cementite) would be 100 per cent cementitic. However, cementite is a metastable compound, and is rather easily decomposed into its constituents, iron and carbon (graphite). Free or massive cementite is found in large amounts only in white and mottled irons. There it usually occurs largely with austenite in a eutectic known as ledeburite. A micrograph of a white iron is shown in Fig. 8. The white portions are cementite. Cementite is very hard (probably well above 550 Brinell hardness) and brittle.

Cementite is completely soluble in molten iron, but only partially soluble in solid iron. The excess is rejected during freezing. The remainder occurs as a solid solution. The solid solution of cementite (below the freezing point and above the pearlitic transformation, that is, above 1340 F.) is known as austenite. The solubility of cementite in saturated austenite is about 0.85 per cent at about 1340 F. up to about 1.7 per cent at the final freezing temperature.

Hence, when an iron containing saturated austenite is cooled it rejects some more cementite. (The exact location of the cementite-austenite solubility curve is not fully agreed upon.) In presence of sufficient silicon and with slow cooling through the range from solidification to well below 1340 F., all the free cementite and in some cases nearly all of the solid solution cementite may be broken down into graphite and iron. Let us consider, however, the case where the free cementite has decomposed and the saturated solid solution has reached 1340 F., retaining about 0.85 per cent carbon (or somewhat less in commercial irons). At this point there will occur a heat generation or recalescence and on passing below 1340 F., the saturated austenite is converted into pearlite. Below 1340 F. the solubility of cementite in iron is negligible and it can no longer exist in solid solutions in iron as austenite. Therefore, it is precipitated in the form of lamellæ in a ferritic matrix. This structure, shown in Fig. 3, consisting of ferritic grains with many cementitic lamellæ, is called pearlite. Pearlite is an eutectoid, a structure of relatively constant composition, forming at a definite temperature as explained previously.

*Recapitulation.*—Carbon in molten iron occurs as dissolved cementite, or iron carbide $Fe_3C$. The solubility of cementite in solid iron is limited. In cast irons, in the range from final solidification to about 1340 F., part of the cementite is rejected and occurs as free cementite. The remainder occurs as a solid solution of iron and cementite, known as austenite. In presence of silicon and with sufficiently slow cooling the free cementite is decomposed, one of the products of the reaction being graphite. If this reaction is complete, we have a gray iron of pearlitic matrix. If incomplete, we have mottled iron.

The amount of graphite present depends on the amount of carbon originally present and on the degree of decomposition of the cementite. If the decomposition of both free and solid solution cementite is complete, we have an iron consisting entirely of ferrite and graphite (neglecting the steadite and inclusions probably present). If the free cementite is decomposed, yet the austenite is saturated when passing through the eutectoid transformation, we have a pearlitic iron, consisting of pearlite and graphite.

If the decomposition of free cementite is partial, mottled iron results. If free cementite is not decomposed at all, we have a white iron. These reactions are well depicted by the iron carbon diagram shown by Sauveur(4).

The size of graphite flakes, other factors equal, depends on cooling rates. Other factors are composition and thermal history.

The distribution of graphite flakes, that is the type of flake formations or groupings, depends a great deal on the position of the alloy in the iron carbon diagram. It has been shown that these formations in general outline bear a distinct relation to the forms of actually or potentially pre-existent cementite.

If it were possible to interpret the chemical analysis of gray cast iron in terms of structural volume analysis of the various components formed, that is, show the components exactly as they exist in the casting with the relative volumes they occupy, we should have something more worth while than chemical analysis as a basis of criticism and comparison. Table I shows the approximate structural compositions of several typical irons. While not strictly accurate, this structural analysis shows that gray cast iron may contain 4 to 66 per cent ferrite plus dissolved iron silicide; none to 85 per cent pearlite; none to 43 per cent cementite; 1.0 to 15 per cent steadite; 0.10 to 0.67 per cent manganese sulfide; 0.06 to 0.21 per cent iron

TABLE II.—PROPERTIES OF COMPOUNDS SHOWN IN TABLE I.

| | SPECIFIC GRAVITY | TENSILE STRENGTH, LB. PER SQ. IN. | ELONGATION IN 2 IN., PER CENT | BRINELL HARDNESS NUMBER | PHYSICAL CONDITIONS OF THE CASTINGS |
|---|---|---|---|---|---|
| Ferrite | 7.86 | 50 000 | 40 | 95 | Iron |
| Iron silicide[a] (Fe Si) | 6.17 | low | none | ...... | 1 per cent silicon forms 3 per cent iron silicide. |
| Pearlite | 7.846 | 120 000 | 15 | 240 | A laminated structure containing 6½ parts soft iron or ferrite to 1 part cementite. |
| Cementite ($Fe_3C$) | 7.66 | 5 000 | none | 550 or more | A compound of iron and carbon containing 6.67 per cent of carbon. |
| Steadite | 7.32 | none | brittle | very hard | Phosphide of iron ($Fe_3P$), and saturated solution of $Fe_3P$ in iron. This substance occurs by volume about 10 times that of the phosphorus content by weight. |
| Manganese sulfide | 4.00 | none | brittle | ...... | 1.73 parts manganese to 1 part sulfur. |
| Iron sulfide | 5.02 | low | brittle | ...... | 1.75 parts iron to 1 part sulfur. |
| Graphite | 2.55 | none | non-coherent | ..... | ...................... |

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[a] Existent largely in solid solution in the ferrite-rich portion. In normal irons is not discernible as a separate component.

sulfide; none to 10.7 per cent graphite, while the specific gravity may vary from 7.06 to 7.66.

Just what this means may be gleaned better by considering some of the properties of these compounds as shown in Table II.

## GENERAL INFLUENCE OF MASS AND THERMAL HISTORY ON STRUCTURAL MAKE-UP

*Mass:*

When reference is made to influence of mass or of section size on cast iron, what is really meant as a rule is influence of cooling rate. Mass itself is not always necessarily a factor from a structural viewpoint. For example, it is possible to take an iron which has a perfectly gray fracture in an ordinary 1.2-in. diameter test bar, pour it into a water-cooled ingot mold, say 2 in. square and as long as the 1.2-in. bar, and get a mottled or a chilled fracture in the heavier mass.

This subject of cooling rates has already been well covered by one author and the following quotation is taken from his work(5):

> The absolute cooling rate depends on a number of factors—for example, *initial temperature*, *final temperature*, and *composition* of the alloy—(the preceding being reflected to some degree in the *specific heat*) the *speed of pouring* the *volume* of the casting, the *area* exposed to cooling, the heat *conductivity* of the metal itself, and of the mold material, and all those forms of energy made apparent in *thermal arrests*.
>
> Cooling rate is not a straight-line function. The factors mentioned above are still further complicated by the fact that "final" temperature is a very complicated phenomenon. For example, the mold is getting hot as the metal cools down. Beside this we do not have uniform cooling over the cross-sectional area of any section. In other words, the varying cooling gradients between conducting medium and casting on one hand, and between various layers of the casting on the other, plus the fact that the cooling rates are curve functions, makes it meaningless to express cooling rate from solidus to last reaction point in degrees per minute (or per second). However, the effect of even slight cooling gradients within castings are of great practical importance. Every foundryman has noticed the sharp "breaks" and "picture frame" effects found in some fractures. These indicate "critical gradients" and these critical points probably are quite sharply defined. The sharpness of the effects produced by these temperature critical gradients is greater in low-silicon, high-carbon irons. The lower carbon irons and nickel irons have less tendency to show such radical changes in structure due to what must be rather slight thermal gradients. Therefore, these latter irons generally are more uniform in their physical properties from section to section of the same casting. The element silicon also has a very sharply defined "chemical critical gradient"—wherein a few points variation may mean either white or gray iron.
>
> From our present purpose it is sufficient and more simple to consider only relative cooling rate. Assuming relatively close initial temperatures, all final

temperatures being room temperatures, and close values for specific heats, the relative cooling rates are roughly proportional to the ratio of volume to surface area. This is a simple mathematical way of expressing a well-known foundry fact—namely, that the cooling rate is proportional to section size. Expressed another way, the heat to be dissipated in cooling depends directly on the volume of metal, and the rate with which the heat can be abstracted depends on the exposed area—that is, the surface.

The only absolutely certain method of determining cooling rate in a given section is by pyrometric record. For many simple shapes made under similar melting and molding conditions the ratio of volume to surface area will give relative rates within reasonable accuracy.

The effect of cooling rate is indicated in Fig. 9, which is arranged from the data of Maurer and Holtzhausen(6). It will be noted that the lines bordering the proportions of carbon and silicon shift to the left as the cooling rate is prolonged. Maurer and Holtzhausen also found that 1.2-in. diameter bars cast in molds heated to 480 to 840 F. possessed the same structure as bars 3 to 3½ in. in diameter cast in cold molds.

Graphitization of free cementite just under the eutectic solidification temperature is rapid in gray iron composition. As the temperature is lowered graphitization becomes progressively less rapid. During this period—below the eutectic solidification and above the pearlitic transformation—there also is further rejection of cementite from the austenite.

Below the pearlitic transformation, graphitization (of the pearlitic cementite) is slow, so that unless cooling rates are quite slow or silicon and carbon unusually high, the normal structure of most irons remains pearlitic. Below 900 F., graphitization in most irons proceeds so slowly that from the foundryman's viewpoint we may well consider 900 F. as marking the end of the graphitization period.

Obviously with heavy massive sections the cooling rate will be slow and the graphitization more complete. This is especially true of the higher carbon high-silicon irons. In these, graphitization in heavy sections frequently proceeds so far that the matrix contains a high percentage of ferrite.

Low-carbon irons with silicon content properly adjusted will retain approximately the same combined carbon, whether they are cast into heavy sections or light sections. Such irons are said to possess a high degree of "quasi-isotropy"—they are little affected in percentage of structural components by changes in cooling rate, or speaking loosely, by changes in mass or section size. However, their physical properties usually are affected by changes in cooling rate or "mass effect." This is due to the fact that a slow cooling rate promotes larger grain structure, even though the structural component percentages are relatively constant. This effect of "mass" is easily detected under the microscope, and is manifest in the strength-section data given in the section on Effect of Section Size.[1]

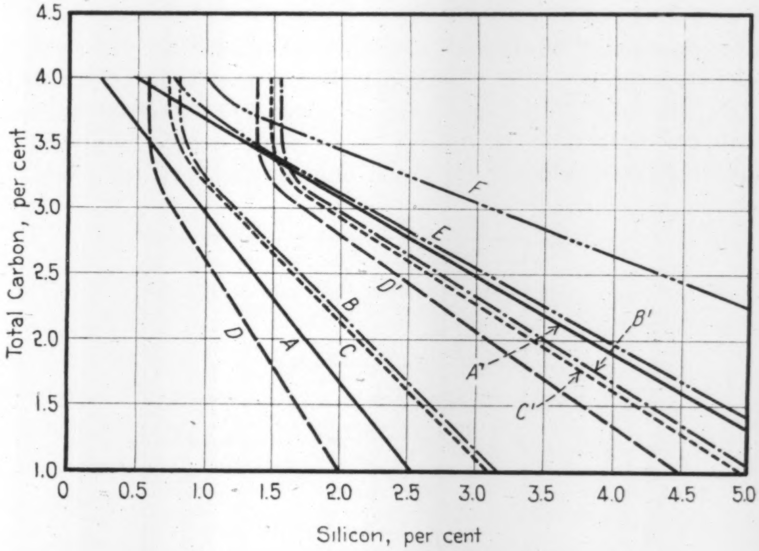

FIG. 9.—Structure of Irons Affected by Cooling Rate.

Line *A-A'* limits of composition for pearlitic structure in bars 1.2 in. in diameter cast in dry sand.

Line *B-B'* limits of composition for pearlitic structures in bars 3 to 3½ in. in diameter cast in chill molds.

Line *C-C'* limits of composition for pearlitic structure in bars 3 to 3½ in. in diameter cast in molds heated to 480 F.

Line *D-D'* limits of composition for pearlitic structure in bars 3 to 3½ in. in diameter cast in molds heated to 840 F.

Line *E*, limit of white iron in bars 3 to 3½ in. in diameter chill cast. (Maurer and Holtzhausen.)

Line *F*, limit of white iron in bars 3 to 4 in. thick chill cast. (Coyle.)

| | MAURER AND HOLTZHAUSEN | COYLE |
|---|---|---|
| Total carbon, per cent | 2.4 to 3.8 | 1.75 to 3.75 |
| Manganese, per cent | 0.8 to 1.0 | under 0.35 |
| Phosphorus, per cent | approx. 0.3 | under 0.01 |
| Sulfur, per cent | approx. 0.1 | under 0.03 |
| Silicon, per cent | 0.25 to 5.1 | 0.30 to 4.2 |

Cooling rates may be retarded artificially by gating as shown by Ronceray(7), by using preheated molds (lately brought out by Lantz, Sipp and Diefenthaler(8), originally tried after the Civil War by New England cannon manufacturers) or accelerated by chills.

---

[1] See p. 190.

*Thermal History:*

The late George K. Elliott(9), in his electric-furnace researches, probably was the first investigator to show definitely that the superheating in itself affected the properties of the iron. He found that in superheated irons more silicon was needed to produce the same effects obtained in cupola irons of otherwise similar analysis.

A number of investigators have studied the phenomena of superheating in detail. These studies led to the formulation of the graphite nuclei hypothesis. The observed facts are that if irons are melted at temperatures within the range of about 2725 to 3100 F., subsequent graphitization on cooling is to some degree inhibited, the graphite flake structures are much finer or smaller than usual, and these structures apparently are not as sensitive to changes in cooling rate (section size) as are the structures of ordinary cupola irons. According to the graphite nuclei theory the superheating completely dissolves the graphite particles contained in the original melting stock. With no nuclei to act as centers of crystallization, graphitization does not commence until the temperature has dropped to a point well under the eutectic. Due to the consequently shorter range for graphitization and the lower temperature at which graphite precipitation is initiated, the flakes are finer and of course there is some apparent carbide stabilization. Figure 10 illustrates the general interrelationship between structure cooling rate and physical properties.

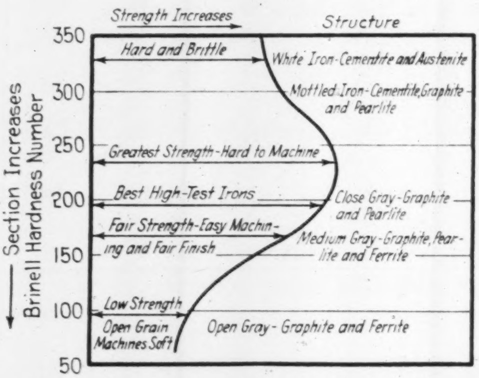

FIG. 10.—Chart Showing General Interrelationship Between Structure, Cooling Rate and Physical Properties. (Bolton and Bornstein.)

High superheat is not readily obtained in cupola melting. It is probable that most cupolas in America average about 2650 F. spout temperature, and comparatively few are able to maintain

2750 F. consistently through a heat. Superheating usually is accomplished by electric furnace heating, either by melting direct or by melting in the cupola and duplexing.

*Other Factors Influencing Structure:*

The character of the original melting stock has a certain degree of influence on the structure of cast iron. A steel addition, for example, usually exhibits an influence by itself as has been noted in comparisons of steel, pig iron, scrap and pig iron-scrap mixes of practically identical analysis. Some claim that this is due to the absence of any graphite in the steel additions.

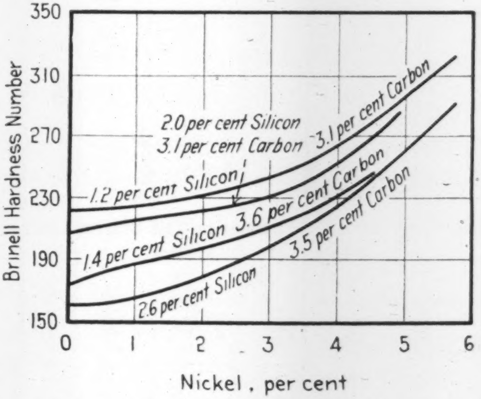

FIG. 11.—Effect of Nickel on the Brinell Hardness of Gray Iron—Arbitration Bar. (P. D. Merica.)

## ALLOY CAST IRONS

All cast irons are alloys. The term "alloy cast iron" is used to describe those cast irons to which such elements as nickel, chromium, molybdenum, etc., have been purposely added because of their effects on the physical properties of the metal. The term "alloy cast iron" is used similarly to the term "alloy steel."

The use of alloying elements such as nickel, chromium, molybdenum, etc., has its place in the production of iron castings. However, these elements should be used for particular purposes and to meet certain physical requirements. Alloy cast irons are frequently used for more severe service than are ordinary cast irons. In the manufacture of alloy steels, particular care is taken in melting practice. Similarly in cast iron, the alloys should be added to a good base metal and not used in an attempt to make a poor iron into a fair one. It should be realized that the large bulk of gray-iron castings are non-alloy castings and most of these castings are satisfactory for the purpose intended.

*Nickel Cast Iron:*

Nickel is usually added to gray cast iron in amounts from 0.25 to 5.0 per cent for general engineering applications. It often is added in combination with other alloying elements, such as chromium or molybdenum. Nickel additions influence machineability, texture, hardness, density, and heat and corrosion-resisting properties. Nickel, like other alloying elements, must be used with care and discretion if economic benefit is to be derived from it. There is no universal formula for nickel cast iron and each problem requires its own solution.

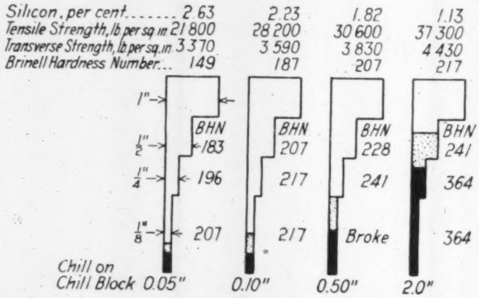
Silicon, per cent........ 2.63 2.23 1.82 1.13
Tensile Strength, lb per sq in 21 800 28 200 30 600 37 300
Transverse Strength, lb per sq. in 3 370 3 590 3 830 4 430
Brinell Hardness Number... 149 187 207 217
1"
$\frac{1}{2}$"
$\frac{1}{4}$"
$\frac{1}{8}$"
BHN
183
196
207
BHN
207
217
217
BHN
228
241
Broke
BHN
241
364
364
Chill on
Chill Block 0.05" 0.10" 0.50" 2.0"

FIG. 12.—Hardening Gray Iron by Decreasing the Silicon Content. The step-bar test. (P. D. Merica.)

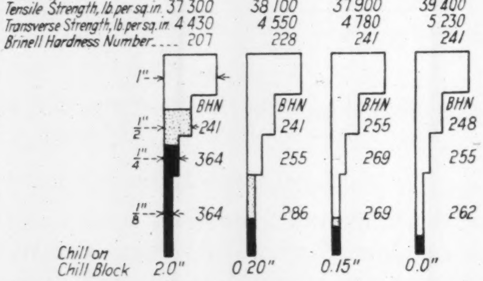
Nickel, per cent.......... 0 1.49 2.12 2.83
Tensile Strength, lb. per sq. in. 37 300 38 100 37 900 39 400
Transverse Strength, lb. per sq. in. 4 430 4 550 4 780 5 230
Brinell Hardness Number.... 207 228 241 241
1"
$\frac{1}{2}$"
$\frac{1}{4}$"
$\frac{1}{8}$"
BHN
241
364
364
BHN
241
255
286
BHN
255
269
269
BHN
248
255
262
Chill on
Chill Block 2.0" 0 20" 0.15" 0.0"

FIG. 13.—Hardening Gray Iron by Adding Nickel. The step-bar test. (P. D. Merica.)

Nickel acts as a graphitizer and does not form a carbide. In small amounts, it will reduce chill and soften partially chilled iron by eliminating hard free carbide, thus promoting machineability. Larger amounts (over 5 per cent nickel) harden iron by forming sorbite and even martensite. See Fig. 11. The value of nickel in iron is primarily related to its effect on machineability. The use of nickel permits of the production of castings, which in comparison with many ordinary iron castings may be:

1. Either harder, stronger, and of better structure with equal machineability, or

2. More readily machineable for the same hardness, strength or structure.

Nickel promotes density and freedom from porosity by permitting the use of lower silicon content without chill or hard spots appearing in light sections. Up to about 4 per cent nickel, in iron of suitable base composition, promotes uniformity of grain, hardness, and strength throughout sections of considerable or uneven thickness.

TABLE III.—TYPICAL EXAMPLES[a] OF NICKEL CAST IRON.

| | CHEMICAL ANALYSIS, PER CENT | | | | | |
|---|---|---|---|---|---|---|
| CASTING | TOTAL CARBON | SILICON | MANGANESE | NICKEL | BRINELL HARDNESS NUMBER | STEEL IN CHARGE, PER CENT |
| Automobile cylinders | 3.25 | 1.80 | 0.65 | 1.25 | 180[b] | 20 |
| High-strength iron | 2.75 | 2.25 | 0.80 | 1.00 | 240 | 90 |
| Locomotive cylinders | 3.00 | 1.10 | 0.85 | 1.25 | 200 | 50 |
| Heavy machine tool beds | 3.20 | 1.00 | 0.80 | 1.00 | 200 | 65 |
| Pump bodies | 3.30 | 1.50 | 0.65 | 1.25 | 200 | 30 |
| Light gears | 3.30 | 1.50 | 0.60 | 1.25 | 200 | 30 |
| Heavy gears | 3.00 | 1.00 | 0.75 | 2.00 | 220 | 50 |
| Automobile pistons | 3.40 | 2.50 | 0.65 | 1.00 | 180 | 15 |
| Heavy crusher frame | 2.75 | 1.75 | 0.80 | 2.00 | 230 | 90 |
| Elevator sheaves | 3.00 | 1.00 | 0.80 | 1.25 | 220 | 50 |

[a] These typical examples should not be used as specifications.
[b] Hardness in bore.

The important relations between silicon content, structure and machineability are recognized by the foundryman. The machineability may be indicated by the step bar casting shown in Fig. 12. The steps of this test bar are fractured and examined and the hardness and combined carbon determined on the various sections. The effect of decreasing silicon content on chill is shown in Fig. 12. In Fig. 13 is shown the effect of nickel in reducing chill. As the nickel is increased, the hardness and strength of the heavier sections increase slightly, and the lighter sections become progressively grayer and more machineable.

Nickel supplements silicon in reducing chill and this permits the use of low-silicon iron in castings, which without the nickel would be difficult to machine. It is by the use of nickel in irons of lower silicon content that the economic benefit of nickel addition to iron may be secured, and simple additions of nickel to iron are not generally economically justified.

The expansion of nickel-alloyed cast irons as compared with that of plain cast iron varies. Up to 5 per cent nickel it is less, then increases until at 20 per cent nickel it is greater than that of plain cast iron; from 20 to 35 per cent nickel it decreases, being the same as that of plain cast iron at 28 to 29 per cent nickel; it increases again above 36 per cent, reaching that of plain iron at 45 to 50 per cent nickel.

As the amount of nickel is increased to from 10 to 18 per cent, there is a gradual change in structure to non-magnetic gamma iron (austenite) with corresponding important changes in properties.

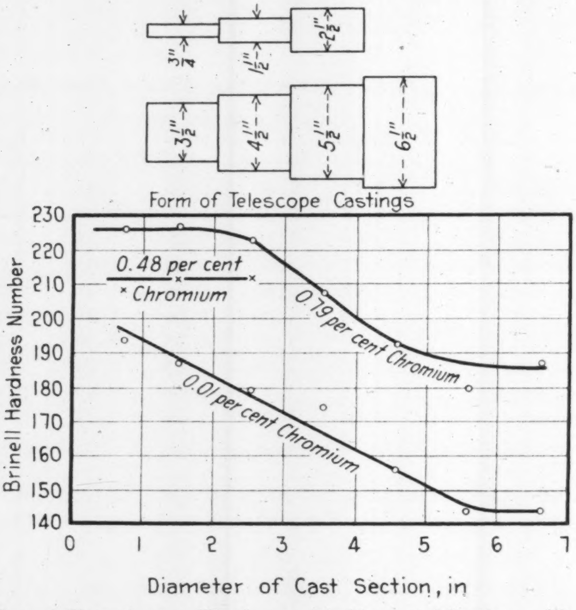

FIG. 14.—Effect of Chromium on Hardness with Change of Section. (C. O. Burgess.)

Austenitic gray irons such as "Ni-Resist" (Example: nickel 14 per cent, copper 6 per cent, chromium 4 per cent, silicon 2 per cent, total carbon 3.0 per cent) are practically free from growth and scaling up to 1500 F. They are resistant to the corrosive action of hydrochloric, sulfuric, acetic, citric, boracic and formic acids, chlorides, sulfates, caustic, brine, sea water, hydrogen sulfide, fuel oil, and other media.

The economic application of nickel to the production of gray iron castings requires a study of individual problems. In Table III are shown some typical examples of nickel cast iron.

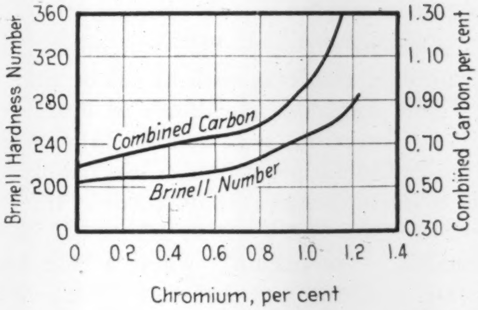

FIG. 15.—Effect of Chromium on Hardness and Combined Carbon of Gray Iron (Arbitration Bars). (Trantin.)

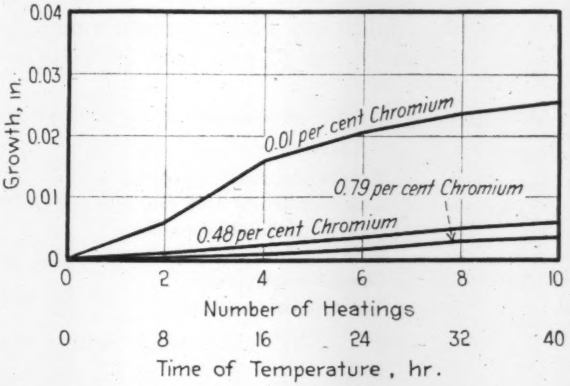

FIG. 16.—Effect of Chromium on Growth of Cast Iron Alternately Heated and Cooled from 1110 F. (C. O. Burgess.)

| | | | | |
|---|---|---|---|---|
| Nickel, per cent | 0 | 0 | 1.1 | 2.1 |
| Chromium, per cent | 0 | 0 | 0.38 | 0 |
| Silicon, per cent | 1.13 | 2.23 | 2.20 | 1.15 |
| Tensile Strength, lb. per sq. in. | 37 300 | 28 200 | 36 300 | 37 900 |
| Transverse Strength, lb per sq. in. | 4 430 | 3 590 | 4 700 | 4 780 |
| Brinell Hardness Number | 207 | 187 | 241 | 241 |

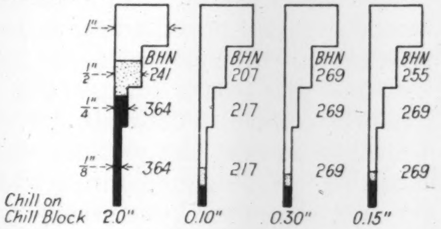
1"
1/2"
1/4"
1/8"
BHN
241
364
364
BHN
207
217
217
BHN
269
269
269
BHN
255
269
269
Chill on
Chill Block
2.0"
0.10"
0.30"
0.15"

FIG. 17.—Hardening Gray Iron by Nickel and Chromium in Balanced Ratio, and Comparison of Nickel-Chromium with Nickel Cast Iron. The step-bar test. (P. D. Merica.)

*Chromium Cast Iron:*

Chromium is essentially a carbide former in contra-distinction to the graphite-forming elements such as silicon and nickel. Chromium increases chill, hardness, resistance to heat, and strength up to certain limits. Chromium iron double carbides are more stable than straight iron carbides and graphitize less readily.

One of the important uses of chromium is the addition of this alloying element to base irons where additional hardness or wear resistance is required. For example, where a base iron in a foundry has a silicon content of 2.25 per cent and a total carbon content of 3.40 per cent. This base iron is suitable for the bulk of the castings produced, but is not suitable for some of the heavier castings and for some medium section castings where a maximum of wear resistance is required. Additions of chromium, ranging from 0.40 to 1.00 per cent frequently are of considerable value. In heavy sections, the use of chromium helps to give a more uniform hardness with change of section as shown in Fig. 14.

TABLE IV.—APPROXIMATE EFFECT OF CHROMIUM ON A SOFT CAST IRON.

| CHROMIUM, PER CENT | STRUCTURE |
|---|---|
| 0 | Ferrite and coarse graphite |
| 0.30 | Less ferrite and finer graphite and pearlite |
| 0.60 | Fine graphite and pearlite |
| 1.00 | Fine graphite and pearlite and small carbides |
| 3.00 | Graphite disappears |
| 5.00 | Much massive carbide |
| 10.00 to 30.00 | Fine carbides |

Table IV shows the approximate effect of chromium on a soft iron.

Chromium additions reduce the graphitic carbon content and decrease the flake size. Chromium favors the formation of more finely laminated and harder pearlite. In a suitable base iron, these effects add to tensile strength, promote finer grain size, and increase hardness. The effect of chromium additions on Brinell hardness and combined carbon of gray iron is shown in Fig. 15.

Above a certain maximum, usually about 1 per cent chromium in soft irons of medium section, free carbides appear with resulting difficulty in machining. Unless graphitizing alloys such as nickel are added, 3 per cent chromium irons usually have entirely white fracture. Higher percentages of chromium may be added to produce special purpose white iron, where freedom from scaling, toughness, or chill is desired. Considerable work is being done on cast irons with chromium contents from 15 to 35 per cent and very good results have been secured in respect to heat and corrosion resistance. For applications up to about 1300 F. (for service comparable to ordinary grate bars) the use of about 1¼ per cent chromium has been valuable in eliminating growth.

Chromium increases the tensile strength at elevated temperatures and minimizes tendency of growth in cast iron. For the effect of chromium on growth of cast iron, see Fig. 16.

The carbide stability conferred by chromium is useful in gray iron heat treatment because the iron does not offset by excessive graphitization the advantages conferred by structural changes in the matrix.

TABLE V.—TYPICAL ANALYSES[a] OF APPLICATIONS OF CHROMIUM CAST IRON.

| CASTING | TOTAL CARBON, PER CENT | SILICON, PER CENT | CHROMIUM, PER CENT |
|---|---|---|---|
| Annealing boxes | 1.80 | 1.25 | 26.0 |
| Chills | 3.10 | 2.00 | 1.0 |
| Crusher jaws and rolls | 2.00 | 1.00 | 25.0 |
| Furnace castings | 2.25 | 2.00 | 15.0 |
| Glass molds | 3.25 | 2.00 | 0.60 |
| Grinding balls and liners | up to 3.5 | up to 2.0 | 3.0 |
| Heat-resistant castings: | | | |
| (*a*) For temperatures up to 1300 F. (such as fire pots and stove grates) | up to 3.5 | 2.25 | 1.25 |
| (*b*) For temperatures to 1800 F. | up to 2.5 | up to 2.50 | 15 to 30 |
| Gears | 3.25 | 1.75 | 0.75 |

---

[a] These compositions *should not* be used for specifications.

In Table V are shown some typical analyses (total carbon, silicon and chromium only), showing applications of chromium cast iron.

*Nickel-Chromium Cast Iron:*

Nickel-chromium gray cast irons are currently produced in a variety of compositions, suited to particular casting or foundry problems for which they are designed. The range generally is not over 3.0 per cent nickel and 1.0 per cent chromium. Nickel and chromium are used together in gray iron primarily for the purpose of refining its structure and of hardening and strengthening it without impairing its machineability.

The addition of about 4.5 per cent nickel and 1.5 per cent chromium to a chilled or white cast iron is being used to give an extremely hard, tough, and strong chilled iron. The hardness of such a material will range from 600 to 750 Brinell as against 380 to 530 Brinell for corresponding grades of plain chilled or white iron.

Nickel and chromium usually are added to gray iron in the proportion in which they mutually neutralize each other in respect to chill. The resulting alloy cast iron will have the same chilling tendency as the plain iron to which the alloys were added, and the casting made from it will be as readily machineable as castings made from the plain base iron. At the same time the refining, matrix hardening and strengthening effects of both alloys will be conserved and reflected in the structure and properties of the casting. Figure 17 illustrates the effect of adding nickel and chromium to gray cast iron.

TABLE VI.—TYPICAL ANALYSES[a] AND APPLICATIONS OF SOME NICKEL-CHROMIUM CAST IRONS.

| | Total Carbon, per cent | Silicon, per cent | Manganese, per cent | Nickel, per cent | Chromium, per cent | Brinell Hardness Number | Steel in Charge, per cent |
|---|---|---|---|---|---|---|---|
| Light automotive cylinders | 3.30 | 2.10 | 0.60 | 0.75 | 0.30 | 200 | 20 |
| Heavy automotive cylinders | 3.30 | 1.85 | 0.65 | 1.50 | 0.60 | 220 | 25 |
| Light forging dies | 3.30 | 1.50 | 0.60 | 1.50 | 0.60 | 300[b] | 30 |
| Heavy forming dies | 3.00 | 1.25 | 0.60 | 2.75 | 0.80 | 300[c] | 50 |
| Brake drums | 3.25 | 2.00 | 0.65 | 1.50 | 0.60 | 220 | 20 |
| Light cylinder liners | 3.25 | 2.00 | 0.60 | 1.50 | 0.60 | 220 | 25 |
| Caustic pots | 3.30 | 0.70 | 0.50 | 1.50 | 0.60 | 230 | 35 |
| Clutch plates | 3.30 | 2.10 | 0.65 | 1.25 | 0.40 | 220 | 15 |
| Permanent molds | 3.25 | 1.75 | 0.60 | 1.50 | 0.60 | 220 | 25 |

---

[a] These typical analyses *should not* be used as specifications.
[b] Heat Treatments: Oil quench from 1575 F. Draw 800 to 1100 F.
[c] Heat Treatments: Oil quench from 1550 F. Draw 800 to 1100 F.

The usual ratio of nickel to chromium for additions to gray iron is $2\frac{1}{2}:1$. This ratio usually gives good results in respect to machineability and physical properties. Other ratios have been used with good results, the relative amounts of nickel and chromium depending on the base iron and the application.

The industrial applications of nickel-chromium cast irons are for castings where better structure, greater hardness, wear resistance or strength are required in machineable iron. The use of nickel and chromium frequently is more convenient than adding nickel alone because it is easier and creates less disturbance in the foundry to add nickel and chromium to the regular base iron than to change the cupola mix (in order to lower the silicon content) and use nickel alone. Nickel and chromium frequently are cheaper than the addition

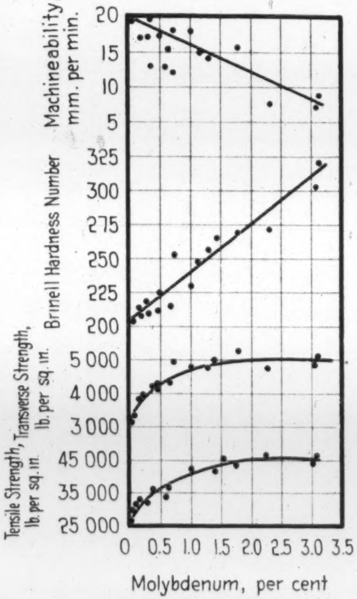

FIG. 18.—Effect of Molybdenum on the Properties of Gray Cast Iron. (Smith and Aufderhaar.)

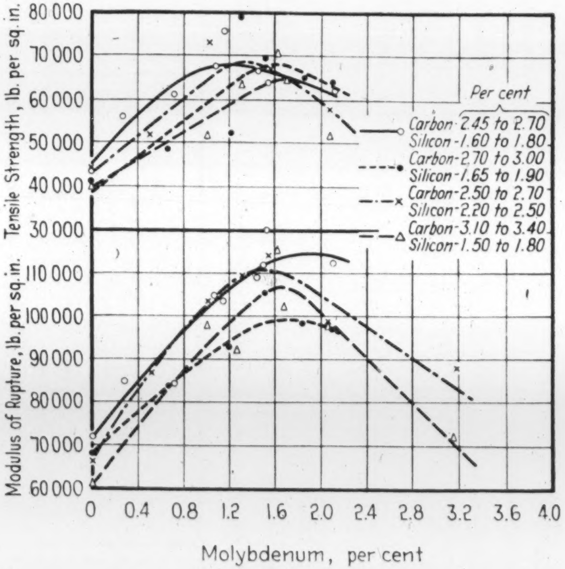

FIG. 19.—Effect of Molybdenum on Strength Properties of Cast Iron. (Musatti and Colbiani.)

of nickel alone. The usual amounts of nickel and chromium added to gray iron to harden and strengthen it vary within the following ranges:

| | NICKEL, PER CENT | CHROMIUM, PER CENT |
|---|---|---|
| Small addition | 0.10 to 0.50 | 0.10 to 0.25 |
| Medium addition | 0.75 to 1.50 | 0.25 to 0.50 |
| Maximum addition | 2.00 to 3.00 | 0.50 to 1.00 |

In Table VI are shown some typical examples of nickel-chromium cast iron.

*Molybdenum Cast Iron:*

Molybdenum in cast iron forms both complex carbides and ferrite solid solution. As a carbide former, it is much less drastic in its action than chromium. It has a beneficial effect on the shape of the graphite flakes, causing them to be somewhat nodular in form with consequent advantage to physical properties.

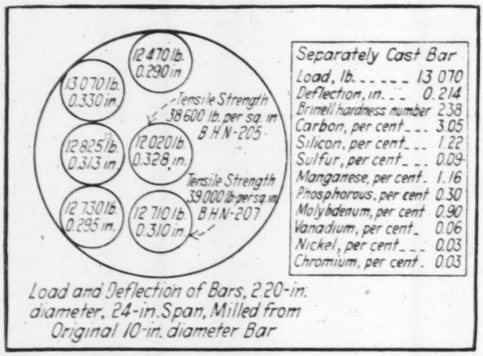
12 470 lb.
0.290 in
13 070 lb.
0.330 in.
Tensile Strength
38 600 lb. per sq. in
B H N-205
12 825 lb.
0.313 in.
12 020 lb.
0.328 in.
Tensile Strength
39 000 lb. per sq. in
B H N-207
12 730 lb.
0.295 in.
12 710 lb.
0.310 in.
Separately Cast Bar
Load, lb. ...... 13 070
Deflection, in. ... 0.214
Brinell hardness number 238
Carbon, per cent ... 3.05
Silicon, per cent ... 1.22
Sulfur, per cent ... 0.09
Manganese, per cent. 1.16
Phosphorous, per cent 0.30
Molybdenum, per cent 0.90
Vanadium, per cent. 0.06
Nickel, per cent ... 0.03
Chromium, per cent. 0.03
Load and Deflection of Bars, 2.20-in. diameter, 24-in. Span, Milled from Original 10-in. diameter Bar

FIG. 20.—Properties of Molybdenum Cast Iron as Influenced by Size of Section. Separately cast bar 2.2 in. in diameter. (MacKenzie.)

The addition of molybdenum to gray cast iron of suitable composition increases tensile strength, transverse strength and deflection, and hardness. Molybdenum usually is added in quantities from 0.25 to 1.25 per cent. Molybdenum is the most effective alloying element added to gray cast iron to increase strength. Up to about 1.5 per cent molybdenum there is an increase of tensile and transverse strengths. Above 2 per cent there is a dropping off in strength. See Figs. 18 and 19.

A number of investigators report that molybdenum increases the wear resistance of cast iron. The addition of molybdenum to cast iron promotes structural uniformity in heavy sections. As a result, good density, Brinell hardness and strength are maintained throughout the cross-section. See Fig. 20.

Molybdenum retards the critical transformation in both the liquid and solid states. This makes the volume changes less drastic and the rate of graphitization slower. Such effects help in heat treating processes, also in the retention of strength at elevated temperatures.

TABLE VII.—TYPICAL ANALYSES[a] OF MOLYBDENUM CAST IRON.

| APPLICATION | TOTAL CARBON, PER CENT | SILICON, PER CENT | MANGANESE, PER CENT | MOLYBDENUM, PER CENT | NICKEL, PER CENT | CHROMIUM, PER CENT |
|---|---|---|---|---|---|---|
| Brake drums | 2.75 | 2.25 | 0.70 | 0.50 | .... | .... |
| Light forging dies | 3.30 | 2.00 | 0.60 | 1.00 | .... | .... |
| Heavy forming dies | 3.10 | 1.50 | 0.60 | 1.00 | .... | .... |
| Pistons, automobile | 3.35 | 2.25 | 0.65 | 0.40 | .... | .... |
| High strength iron | 2.80 | 2.20 | 0.80 | 0.35 | .... | .... |
| Automobile cylinders | 3.20 | 2.10 | 0.60 | 0.35 | .... | .... |
| Automobile cylinders | 3.20 | 2.30 | 0.60 | 0.35 | .... | 0.30 |
| Ingot molds | 3.50 | 1.20 | 0.75 | 0.50 | .... | .... |
| Automotive castings, high strength | 3.00 | 2.35 | 0.80 | 0.50 | 0.40 | 0.40 |

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[a] These are typical analyses only and *should not* be used as specifications.

TABLE VIII.—TYPICAL ANALYSES[a] OF VANADIUM CAST IRON.

| | TOTAL CARBON, PER CENT | SILICON, PER CENT | MANGANESE, PER CENT | SULFUR, PER CENT | PHOSPHORUS, PER CENT | VANADIUM, PER CENT | CHROMIUM, PER CENT | NICKEL, PER CENT |
|---|---|---|---|---|---|---|---|---|
| Grate bars | 3.68 | 1.72 | 0.53 | 0.09 | 0.43 | 0.14 | .... | .... |
| Locomotive cylinders | 3.47 | 1.54 | 0.53 | 0.10 | 0.49 | 0.13 | .... | .... |
| Forming dies | 3.40 | 1.30 | 0.50 | 0.08 | 0.23 | 0.12 | .... | .... |
| Steel mill rolls | 3.50 | 1.10 | 1.20 | 0.10 | 0.20 | 0.35 | 1.85 | 4.00 |
| Bottle molds | 3.30 | 1.65 | 0.60 | 0.07 | 0.30 | 0.14 | .... | .... |

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[a] These are typical analyses only and *should not* be used as specifications.

There is conflicting evidence on the effect of molybdenum on machineability. When properly used, suitable machineability can be obtained. Molybdenum probably has little influence on the corrosion resistance of cast iron. Molybdenum can be used with or without other alloying elements in cast iron. There are applications of molybdenum with chromium, nickel and nickel-chromium.

In Table VII are given some typical analyses of molybdenum cast iron.

### *Vanadium Cast Iron:*

Vanadium is added to cast iron in amounts varying from about 0.10 to about 0.50 per cent. The following effects are observed:

1. Interference with graphitization; that is, stabilization of cementite.
2. Smaller graphite flakes and more uniform graphite distribution.
3. A tendency to promote sorbitic structure.
4. Decreased grain size of matrix.
5. Smaller and less pronounced and less perfectly aligned dendrites in the surface layers of chilled castings. Due to this and the greater stabilization of cementite, greater depth, hardness, toughness and wear resistance of chill.
6. Resistance of the carbide to decomposition at elevated temperatures.

These results of vanadium additions are largely a carbide-stabilizing effect. Vanadium is the most powerful of the carbide-forming elements. It lowers the eutectoid ratio more rapidly than any other element and gives evidence of forming stable carbides at moderately elevated temperatures.

Vanadium cast iron is used for a number of purposes, the following analyses in Table VIII being typical.

### *Copper in Cast Iron:*

Copper cometimes is used in quantities up to 1.0 per cent to impart corrosion resistance to cast iron. Copper is particularly efficient in combating corrosion by water, dilute acids and dilute salt solutions. Copper increases toughness and Brinell hardness. Also, it slightly improves machineability.

### *Titanium Cast Iron:*

Titanium has a graphitizing influence on cast iron similar to silicon but stronger than that element in small amounts. The effect on graphitization involves an increase in ferrite formation and a decrease in the chilling tendency when titanium is added. The deoxidizing and scavenging affects of titanium on cast iron have been reported. Titanium also is used in combination with chromium in cast iron, giving a refining action on the grain.

### *Aluminum Cast Iron:*

Aluminum acts as a strong graphitizer in low-silicon irons. Small amounts of aluminum or aluminum-silicon alloys have a strong tendency to graphitize white iron. Between 1.0 and 1.75 per cent aluminum is effective in producing extreme hardness in the nitrided case of cast iron. A disadvantage is the fact that when aluminum cast iron is poured, a tenacious oxide skin forms on the surface, getting thicker as the pouring proceeds, and finally tending to form unsound places in the casting.

*Zirconium Cast Iron:*

In an iron of suitable composition, with low silicon, zirconium additions improve physical properties. In amounts from 0.10 to 0.30 per cent, zirconium deoxidizes cast iron and raises strength and resistance to impact.

Small amounts of zirconium-silicon alloys have a strong tendency to graphitize white irons.

## III. PROPERTIES OF CAST IRON

### SUMMARY OF PROPERTIES

Cast iron is widely used in engineering and allied industries because of the ease with which it may be cast sound, its low cost, and its wide range of physical properties. The attainable properties are not generally appreciated and frequently are subordinated to get low cost, with conscious or unconscious sacrifice in utility. This is due to incomplete understanding of the properties obtainable and to some degree to a lack of proper test methods. During the past decade this situation has been rapidly changing. In this section the ranges of properties of the series of alloys under gray and white cast irons are summarized. In subsequent sections, to which references are given, will be found detailed discussions of these ranges:

*Tensile Strength.*—Tensile strength is regarded as the most important property. When tests are correctly made, the results are reliable and comparable. For gray cast irons the tensile strength can be controlled within close limits over a wide range, the range being from under 20,000 lb. per sq. in. to over 60,000 lb. per sq. in. This is further elaborated under Mechanical Properties, see page 169.

*Compression.*—Usually the compressive strength is three to five times the tensile strength, the narrower ratios applying to the stronger irons and the wider to the weaker irons. For gray irons the range would be from about 70,000 to 200,000 lb. per sq. in., as discussed in greater detail under Mechanical Properties, see page 176.

*Transverse Strength and Deflection.*—The transverse test is the most commonly used test for cast irons. Its principal use is as a foundry control test. As indicated in the section on Modulus of Rupture,[1] the transverse modulus of rupture is much higher than the tensile strength. For gray irons, 1.2-in. diameter bar tested on 18-in. centers, the range of breaking load will be about 1300 lb. to 4200 lb., with deflections ranging from 0.16 to 0.40 in. Detailed data and discussion are presented under Mechanical Properties, page 170, and under Classification and Specifications, page 239.

*Elastic Properties.*—Gray cast iron does not follow Hooke's law exactly. The effective modulus of elasticity at 25 per cent of the ultimate strength ranges from 12,000,000 lb. per sq. in. for the weaker irons to 18,000,000 lb. per sq. in. or better for the strong types of gray iron. There is no well-defined elastic limit. Gray irons can sustain static loads up to 80 per cent or more of their tensile strength without failure. The section on Elasticity, Plasticity, Stiffness, page 180, discusses this in greater detail.

*Shear Strength.*—Shear strength will be from about 1.0 to 1.6 times the tensile strength, the narrower ratio applying to the higher strength irons and the wider ratio to the weaker irons. For gray irons the shear strength will range from about 25,000 to 80,000 lb. per sq. in. See section on Shear Strength under Mechanical Properties, page 177, for additional data and discussion.

*Fatigue or Endurance Limit.*—The endurance limit varies from about 42 to 57 per cent of the tensile strength, depending on the type of iron. For the more common gray irons this would be approximately from about 9000 lb. per sq. in. to about 26,000 lb. per sq. in. Special high-test irons would run proportionately higher. Fatigue Properties of Cast Iron are elaborated on page 178.

*Impact or Shock Resistance.*—Cast iron is not used commonly to resist impact or shock, yet as between different cast irons there are great differences in impact strength. There is no definite relationship between shock resistance and tensile or transverse strength. In general, however, it may be stated that with increase in tensile and transverse strengths, there is an increase in shock resistance. Impact resistance depends largely on the form and quantity of graphite present. A detailed discussion of impact testing and shock resistance appears on page 184.

*Hardness.*—Hardness can be controlled within certain limits by method of manufacture, composition and rate of solidification. A wider range of hardness is possible by heat treatment. The hardness range obtainable in cast iron varies from slightly over 100 Brinell for fully annealed cast iron to almost 450 for ordinary chilled irons and up to 700 for certain special chilled and heat-treated irons. Further data in respect to hardness are given on pages 189, 247 and 250.

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[1] See p. 170.

*Heat Resistance.*—Ordinary cast irons will retain their strength up to about 850 F. Special alloy irons have been developed to resist heat effects up to about 1800 F. See page 227 for data and detailed discussion of heat resistance.

*Wear Resistance.*—Gray cast iron has exceptionally good wearing properties, being especially free from tendency to gall or seize. This is due in part to the self-lubricating action of the graphite flakes. Where resistance to abrasion is required, the hardness of the white or chilled irons is a desirable property. Wear resistance is discussed in detail on pages 212 and 247.

*Machineability.*—The composition, structure and physical properties of cast irons extend over a wide range and, dependent upon these factors, the machineability of cast iron varies from being one of the most readily machineable to the most difficult among ferrous metals. Ease of machining is usually inversely proportional to the strength of the casting. Effects of chilling, treatment by special alloys, heat treating and special methods of manufacture may modify this statement. The section on Machineability of Cast Iron, page 206, gives a complete discussion of this property.

*Corrosion Resistance.*—Under normal conditions plain cast irons are comparatively corrosion resistant. For more severe conditions alloyed cast irons have been developed and are proving of exceptional value in many fields of application where corrosion is an important factor. Corrosion resistance is discussed in greater detail on page 215.

*Mass Effect.*—Mechanical properties—tensile, compressive, fatigue and hardness—vary considerably with change of section thickness. Modern methods of testing take this variable into consideration. Irons produced by foundries with adequate metallurgical control vary less in properties from section to section than irons formerly used. This is especially true of the higher strength pearlitic-base irons. For further discussion of mass effect and effect of section size, see pages 141 and 190.

*Electrical and Magnetic Properties.*—Electrical and magnetic properties are discussed on pages 167 and 168.

*Specific Gravity.*—The specific gravity will vary from about 7.00 for a coarse-grained cast iron to about 7.70 for a white cast iron. In terms of weight per cubic foot and cubic inch this would be approximately 437 to 487 lb. per cu. ft. and 0.26 to 0.29 lb. per cu. in. Table I and the section on Specific Gravity, page 165, give further data on this subject.

*Heat Effects.*—(*a*) *Melting Range.*—Cast irons do not have a definite melting point due to varied structural components. For gray irons with high phosphorus content the melting of the steadite may begin at about 1750 F. For ordinary gray irons the melting range will vary from about 2000 to 2400 F., as discussed under Melting and Freezing Ranges on page 162.

(*b*) *Specific Heat.*—The specific heat of cast iron at ordinary temperatures can be assumed as the average of its constituents, as discussed on page 162.

(*c*) *Thermal Conductivity.*—Thermal conductivity decreases with rising temperatures. For gray irons between 50 to 450 F. it will vary from about 290 to 360 B. t. u. per hr. per sq. ft. per in. of thickness per 1 F. of temperature difference, or 0.108 to 0.149 cal. per sq. cm. per 1 C. Detailed data appear on page 166.

(*d*) *Solid Contraction or Pattern Makers' Shrinkage.*—The commonly used figure for pattern makers' shrinkage for gray cast iron is $\frac{1}{8}$ in. per ft. and for white iron $\frac{1}{4}$ in. per ft. These figures will not be sufficient for very accurate work, as composition of the metal and type and size of pattern will cause variations. For further discussion on volume change in castings and on shrinkage, etc., see pages 164 and 261.

TABLE IX.—MELTING AND FREEZING RANGES OF TYPICAL CAST IRONS.

| | IRON No. 1 | IRON No. 2 | IRON No. 3 | IRON No. 4 | IRON No. 5 | IRON No. 6 | IRON No. 7 | IRON No. 8 |
|---|---|---|---|---|---|---|---|---|
| Liquidus, deg. Fahr. | 2102 | 2102 | 2102 | 2111 | 2165 | 2282 | 2282 | 2462 |
| deg. Cent. | 1150 | 1150 | 1150 | 1155 | 1195 | 1250 | 1250 | 1350 |
| Carbon, per cent... | 3.72 | 3.66 | 3.50 | 3.27 | 2.90 | 3.08 | 3.10 | 2.24 |
| Silicon, per cent.... | 2.10 | 2.87 | 2.86 | 2.87 | 2.88 | 1.68 | 1.69 | 1.24 |
| Phosphorus, per cent | 0.46 | 0.68 | 0.67 | 0.59 | 0.66 | 0.35 | 0.35 | 0.27 |

(*e*) *Coefficient of Thermal Expansion.*—At room temperatures the commonly used value of 0.000010 per 1 C. is sufficiently accurate for ordinary temperature changes. See section on Coefficient of Expansion, page 165.

(*f*) *Heat Resistance, Growth, and Creep.*—Ordinary cast irons are serviceable when used at temperatures below 650 F., but under certain conditions growth begins above this temperature. Alloy heat-resisting cast irons have been developed to withstand operating temperatures up to 1800 F. Reference should be made to the sections on Effects of Elevated Temperatures, page 197, Properties of Gray Iron at Sub-Normal Temperatures, page 202, Growth of Iron, page 203, and Heat Resistance of Cast Iron, page 227.

TABLE X.—FREEZING POINT OF SOME IRONS.

| Freezing Point, Deg. Fahr. | Freezing Point, Deg. Cent. | Carbon, per cent | Silicon, per cent | Phosphorus, per cent | Nickel, per cent | Chromium, per cent | Copper, per cent | Molybdenum, per cent |
|---|---|---|---|---|---|---|---|---|
| 2021 | 1105 | 3.25 | 2.08 | 1.99 | .... | .... | .... | ...... |
| 2071 | 1133 | 3.61 | 2.06 | 0.75 | .... | .... | .... | ...... |
| 2079 | 1137 | 3.53 | 1.67 | 0.68 | .... | .... | .... | ...... |
| 2082 | 1139 | 3.41 | 2.44 | 0.63 | .... | .... | .... | ...... |
| 2084 | 1140 | 3.49 | 1.68 | 0.74 | .... | .... | .... | ...... |
| 2088 | 1142 | 3.07 | 1.97 | 0.40 | .... | .... | .... | ...... |
| 2093 | 1145 | 3.08 | 2.07 | 0.04 | 16.29 | 1.89 | 6.12 | ...... |
| 2107 | 1152 | 3.49 | 2.08 | 0.19 | 1.17 | 0.65 | 0.32 | ...... |
| 2116 | 1158 | 3.95 | 1.90 | 0.07 | .... | .... | 0.32 | ...... |
| 2125 | 1163 | 3.79 | 1.47 | 0.12 | .... | .... | 0.21 | ...... |
| 2127 | 1164 | 3.52 | 2.94 | 0.04 | .... | .... | 0.34 | ...... |
| 2127 | 1164 | 3.46 | 2.35 | 0.15 | 0.65 | .... | .... | ...... |
| 2138 | 1170 | 2.79 | 2.44 | 0.03 | 0.50 | 0.09 | .... | (Treated with Calcium-Silicide) |
| 2142 | 1172 | 3.38 | 1.18 | 0.14 | .... | .... | .... | ...... |
| 2142 | 1172 | 2.88 | 1.99 | 0.43 | .... | .... | .... | ...... |
| 2142 | 1172 | 3.43 | 2.35 | 0.20 | .... | .... | .... | ...... |
| 2143 | 1173 | 3.14 | 2.69 | 0.18 | 0.25 | 0.09 | .... | 0.07 |
| 2145 | 1174 | 3.12 | 2.18 | 0.63 | .... | .... | .... | ...... |
| 2147 | 1175 | 3.39 | 2.50 | 0.43 | .... | .... | .... | ...... |
| 2151 | 1177 | 3.25 | 1.63 | 0.42 | .... | .... | .... | ...... |
| 2156 | 1180 | 3.49 | 0.53 | 0.33 | .... | .... | .... | ...... |
| 2158 | 1181 | 3.05 | 2.70 | 0.16 | 0.60 | 0.24 | .... | 0.45 |
| 2217 | 1214 | 2.61 | 2.38 | 0.06 | 1.08 | 0.09 | .... | ...... |
| 2224 | 1218 | 2.50 | 2.20 | 0.04 | .... | 0.08 | .... | ...... |
| 2233 | 1223 | 2.53 | 3.25 | 0.02 | 10.59 | 1.55 | 4.07 | ...... |

TABLE XI.—SPECIFIC HEAT VALUES AT VARIOUS TEMPERATURES.

| Temperature Deg. Fahr. | Temperature Deg. Cent. | Average Specific Heat |
|---|---|---|
| 212 | 100 | 0.131 |
| 392 | 200 | 0.134 |
| 572 | 300 | 0.137 |
| 752 | 400 | 0.140 |
| 932 | 500 | 0.142 |
| 1112 | 600 | 0.148 |
| 1292 | 700 | 0.154 |
| 1472 | 800 | 0.168 |
| 1652 | 900 | 0.172 |
| 1832 | 1000 | 0.175 |
| 2012 | 1100 | 0.178 |
| 2192 | 1200 | 0.219 |
| 2372 | 1300 | 0.218 |

(*g*) *Heat Treatment.*—Cast irons respond to heat treatments such as hardening and tempering, nitriding, annealing to remove strains, or annealing to soften for improved machineability. A detailed discussion in respect to heat treatment appears on page 240.

## PHYSICAL CONSTANTS

### *Melting and Freezing Ranges:*

Saeger and Ash(10) determined the liquidus for their investigation of volume changes and obtained the results given in Table IX on eight cast irons (the last of which was mottled).

They found that 2102 F. (1150 C.) was the temperature of the solidus for all these irons.

Roeser and Spencer measured the practical freezing point of the twenty-five irons covered in the Report on the Impact Investigation of A.S.T.M. Committee A-3(2), recently completed. This freezing point was taken as the point at which the pyrometer gave at least four successive readings of the same value at 15-second intervals in a casting 4 by 4 by 8 in., molded in sand, open top, or in a few cases where there was no defined freezing point the thermocouple was removed when the metal began to seize the tube. Their results are given in Table X, the irons being listed in order of increasing temperature of freezing.

The melting point of gray cast iron lies somewhat above the freezing point. This difference is a function of the rate of heating and the size of the graphite. A gray cast iron, if it could be immediately brought to the melting point, would have the melting point of a steel with carbon content equal to the combined carbon of the cast iron (also the same silicon, phosphorus, etc.). Since no such heating is possible in the foundry, some of the graphite redissolves before melting is accomplished. The faster the heating and the larger the graphite flakes, the less solution takes place, therefore the higher the melting point. Moldenke actually proved this by melting pigs of gray and white iron side by side in an open-hearth furnace. He observed that "white irons begin to melt before gray irons of similar section every time."

### *Specific Heat:*

The specific heat at ordinary temperatures can be assumed as the average of its constituents. Umino gives for a pig iron containing 4.22 per cent carbon, 1.48 per cent silicon, 0.12 per cent phosphorus, 0.73 per cent manganese and 0.03 per cent sulfur, the valuation between temperature and specific heat as shown in Table XI.

The heat of fusion, Umino give as 47 cal. per g.

## TABLE XII.—DATA ON VOLUME CHANGE.[a]

| | No. VIII | | No. VII | | No. I | | No. IV | | No. IX | | No. XIII | | No. II | | No. III | | No. XIV | | No. VI | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar | 1½-in. Bar | ¾-in. Bar |
| Linear contraction from 2102 F. (1150 C.), per cent | 0.67 | 1.08 | 0.83 | 1.08 | 0.93 | 1.13 | 0.95 | 1.11 | 0.97 | 1.08 | 0.98 | 1.09 | 1.06 | 1.11 | 1.08 | 1.12 | 1.12 | 1.17 | 1.31 | 1.77 |
| Linear contraction from 1958 F. (1070 C.), per cent | 0.90 | 1.17 | 1.04 | 1.14 | 1.13 | n.d. | n.d. | 1.22 | 1.12 | 1.19 | 1.03 | 1.13 | 1.24 | 1.25 | 1.19 | n.d. | 1.17 | 1.21 | n.d. | n.d. |
| Shrinkage during solidification, per cent | 0.07 | 1.65 | 0.62 | 1.11 | n.d. | n.d. | 1.94 | 1.73 | 0.82 | 1.10 | 0.07 | 1.31 | n.d. | n.d. | 1.59 | 1.94 | 0.00 | 0.07 | 5.82 | 4.85 |
| Liquid contraction per 100 C. (212 F.), per cent | 1.1 | | 1.1 | | n.d. | | 1.1 | | 1.1 | | 1.1 | | n.d. | | 1.1 | | 1.1 | | 1.1 | |
| Density at room temperature, g. per cu. cm | 7.02 | 7.01 | 7.09 | 7.10 | 7.10 | 7.13 | 7.26 | 7.29 | 7.03 | 7.04 | 7.08 | 7.08 | 7.12 | 7.12 | 7.27 | 7.31 | 7.14 | 7.15 | 7.66 | 7.70 |
| Density at 2102 F. (1150 C.), g. per cu cm | 6.89 | 6.80 | 6.90 | 6.85 | 6.90 | 6.89 | 7.04 | 7.04 | 6.85 | 6.80 | 6.89 | 6.80 | 6.90 | 6.89 | 7.04 | 7.04 | 6.90 | 6.90 | 7.35 | 7.30 |
| Density at liquidus, g. per cu. cm | 6.90 | | 6.94 | | n.d. | | 6.92 | | 6.89 | | 6.89 | | n.d. | | 6.92 | | 6.89 | | 6.95 | |
| Liquidus temperature:<br>deg. Fahr<br>deg. Cent | <br>2102<br>1150 | | <br>2102<br>1150 | | n.d. | | <br>2282<br>1250 | | <br>2102<br>1150 | | <br>2111<br>1155 | | n.d. | | <br>2282<br>1250 | | <br>2183<br>1195 | | <br>2462<br>1350 | |
| Total carbon, per cent | 3.69 | 3.63 | 3.67 | 3.76 | 3.63 | 3.63 | 3.10 | 3.10 | 3.39 | 3.60 | 3.27 | 3.27 | 3.71 | 3.71 | 3.08 | 3.08 | 2.90 | 2.89 | 2.18 | 2.29 |
| Graphitic carbon, per cent | 3.53 | 3.42 | 3.26 | 3.35 | 2.97 | 2.99 | 2.31 | 2.27 | 3.20 | 3.40 | 2.88 | 2.90 | 2.99 | 2.96 | 2.18 | 2.23 | 2.68 | 2.65 | 0.56 | 0.42 |
| Silicon, per cent | 2.87 | | 2.10 | | 2.08 | | 1.69 | | 2.86 | | 2.87 | | 2.11 | | 1.68 | | 2.88 | | 1.24 | |
| Phosphorus, per cent | 0.68 | | 0.46 | | 0.44 | | 0.35 | | 0.67 | | 0.59 | | 0.46 | | 0.35 | | 0.66 | | 0.27 | |
| Manganese, per cent | 0.59 | | 0.54 | | 0.50 | | 0.48 | | 0.58 | | 0.52 | | 0.50 | | 0.44 | | 0.44 | | 0.35 | |
| Sulfur, per cent | 0.03 | | 0.05 | | 0.05 | | 0.04 | | 0.03 | | 0.03 | | 0.05 | | 0.04 | | 0.03 | | 0.04 | |

[a] In this table "n.d." denotes "no determination."

Roeser(11), measuring the heat losses from a hot metal car, arrives at a specific heat of 0.23 for pig iron at 2462 F. (1350 C.). International Critical Tables give specific heat between 0 and 100 C. (32 and 212 F.) as 0.13.

*Fluidity:*

Thielmann and Wimmer(12) give the following data for the viscosity of the iron carbon system (silicon 0.1 per cent, manganese 0.5 per cent, phosphorus 0.12 per cent, sulfur 0.06 per cent):

| | CENTIPOISES | | | |
|---|---|---|---|---|
| | 2282 F.<br>(1250 C.) | 2372 F.<br>(1300 C.) | 2462 F.<br>(1350 C.) | 2552 F.<br>(1400 C.) |
| 2.5 per cent carbon......... | .... | .... | 2.7 | 2.3 |
| 3.0 per cent carbon......... | .... | 2.8 | 2.4 | 2.0 |
| 3.5 per cent carbon......... | 2.9 | 2.4 | 2.0 | 1.8 |
| 4.0 per cent carbon......... | 2.1 | 1.8 | 1.6 | 1.5 |

These figures show clearly the effect of temperature and carbon content in increasing the fluidity. A recent paper by Berger(13) shows that the fluidity increases linearly to within a few tenths per cent of the eutectic, rises abruptly to the eutectic and then drops sharply away from it. Spencer(14) had suggested this before, but Berger shows it clearly.

Fluidity seems to be practically given by the formula:

$$\text{Fluidity} = \frac{\text{Pouring Temperature} - \text{Freezing Point}}{\text{Freezing Point} - \text{Mold Temperature}}$$

*Volume Change in Casting:*

The subject is complicated by the influence of cooling rate (usually due to mold conditions or section of metal) on the liberation of graphite—the more graphite the less net change in volume from freezing point to room temperature.

Saeger and Ash(10) give the most complete and authoritative data available on these properties, as shown in Table XII.

It will be noted that there is a marked expansion in all these irons from the freezing point at 2102 F. (1150 C.) down to 1958 F. (1070 C.). This is not all due to graphitization as some expansion occurs at this temperature in white iron also. The authors attribute it tentatively to the evolution of gas.

Attention should be drawn to the effect of these phenomena in the design of castings. When different sections are joined rigidly they must, of necessity, create strains, since they have different cooling rates and arrive at different points on the contraction curves at the same time. Add to this the effect of the greater graphitization in the large section and it will be evident that some designs require the highest degree of skill on the part of the foundryman to keep them from tearing themselves to pieces.

### *Specific Gravity:*

Specific gravity is tabulated for the different important temperatures in Table XII as densities in grams per cubic centimeter.

### *Coefficient of Expansion:*

For the temperature range 1070 C. (1958 F.) to room temperature the coefficient may be read from Table XII by dividing the linear contraction in per cent from 1070 C. (1958 F.) to room temperature (20 C., 68 F.) by 105,000 and it will be seen to vary from 0.0000092 to 0.0000169 per 1 C. At room temperature the commonly used figure of 0.000010 per 1 C. is sufficiently accurate for ordinary temperature changes. Driessen gives the following relation between carbon and coefficient of expansion on annealed samples of low silicon iron (0.1 per cent silicon):

| | Carbon, 2.24 per cent | Carbon, 3.66 per cent | Carbon, 3.80 per cent |
|---|---|---|---|
| Mean coefficient of expansion at 100 C. (212 F.) | 0.0000096 | 0.0000086 | 0.0000087 |
| Mean coefficient of expansion at 200 C. (390 F.) | 0.0000096 | 0.0000088 | 0.0000085 |

### *Surface Tension:*

Draht and Sauerwald(15) determined the surface tension of molten cast iron as given below:

| Temperature | | Specific Gravity | Surface Tension, dynes per sq. cm. |
|---|---|---|---|
| Deg. Cent. | Deg. Fahr. | | |
| 1267 | 2313 | 6.81 | 936 |
| 1277 | 2331 | 6.80 | 938 |
| 1310 | 2390 | 6.75 | 917 |
| 1318 | 2404 | 6.74 | 914 |

| | Chemical Analysis, per cent | | Chemical Analysis, per cent |
|---|---|---|---|
| Carbon | 3.22 | Manganese | 0.56 |
| Silicon | 2.76 | Sulfur | 0.13 |
| Phosphorus | 0.49 | | |

This compares with 1018 for gold at 1120 C. (2050 F.) and 1178 for copper at 1150 C. (2100 F.). The atmosphere was not stated in the reference, but this is of profound influence as a tough oxide film is very troublesome in casting.

*Thermal Conductivity:*

Donaldson(16, 17) reported on the cast irons shown in Table XIII which is sufficiently representative to indicate the values of thermal conductivity to be expected.

TABLE XIII.—THERMAL CONDUCTIVITY OF CAST IRONS.

| | Iron S.1[a] | Iron S.6 | Iron S.8 | Iron S.9 | Iron M.3 | Iron M.4 | Iron Cr.1 | Iron Cr.2 | Iron N | Iron V | Iron W | Iron I.M.1[b] | Iron I.M.2[b] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CHEMICAL COMPOSITION, PER CENT | | | | | | | | | | | | | |
| Total carbon | 3.35 | 3.08 | 3.16 | 3.25 | 3.25 | 3.32 | 3.19 | 3.17 | 3.16 | 3.19 | 3.02 | 3.77 | 3.49 |
| Combined carbon | 0.91 | 0.79 | 0.68 | 0.60 | 0.74 | 0.77 | 0.70 | 0.93 | 0.67 | 0.70 | 0.78 | 0.82 | 0.77 |
| Graphitic carbon | 2.44 | 2.29 | 2.48 | 2.65 | 2.51 | 2.55 | 2.49 | 2.24 | 2.50 | 2.49 | 2.24 | 2.95 | 2.72 |
| Silicon | 0.65 | 1.24 | 1.48 | 1.91 | 1.56 | 1.52 | 1.42 | 1.40 | 1.56 | 1.45 | 1.89 | 1.81 | 2.07 |
| Sulfur | 0.12 | 0.08 | 0.05 | 0.07 | 0.03 | 0.01 | 0.05 | 0.04 | 0.10 | 0.08 | 0.06 | 0.10 | 0.04 |
| Phosphorus | 0.17 | 0.36 | 0.70 | 0.81 | 0.69 | 0.71 | 0.70 | 0.69 | 0.67 | 0.69 | 0.68 | 0.04 | 0.04 |
| Manganese | 0.85 | 0.94 | 0.97 | 0.97 | 1.87 | 2.43 | 0.96 | 0.97 | 0.94 | 0.99 | 0.76 | 0.43 | 0.74 |
| Alloy | .... | .... | .... | .... | .... | .... | 0.20 | 0.39 | 0.75 | 0.12 | 0.48 | .... | .... |
| THERMAL CONDUCTIVITY, *K*, CAL. PER CM. PER SEC. | | | | | | | | | | | | | |
| At 100 C. (212 F.) | 0.135 | 0.127 | 0.122 | 0.111 | 0.119 | 0.117 | 0.127 | 0.131 | 0.108 | 0.122 | 0.118 | 0.094 | 0.112 |
| At 200 C. (390 F.) | 0.128 | 0.122 | 0.117 | 0.108 | 0.114 | 0.112 | 0.122 | 0.126 | 0.106 | 0.117 | 0.114 | 0.092 | 0.100 |
| At 300 C. (570 F.) | 0.122 | 0.116 | 0.113 | 0.105 | 0.109 | 0.106 | 0.116 | 0.120 | 0.104 | 0.112 | 0.109 | 0.089 | 0.108 |
| At 400 C. (750 F.) | 0.114 | 0.109 | 0.107 | 0.103 | 0.104 | 0.101 | 0.111 | 0.115 | 0.101 | 0.107 | 0.104 | 0.087 | 0.106 |

[a] Perlit iron (cast in a hot sand mold).
[b] Sections of ingot molds.

TABLE XIV.—THE EFFECT ON THERMAL CONDUCTIVITY OF ANNEALING AT 550 C. (1020 F.), SHOWN ON IRON S8.

| TREATMENT | COMBINED CARBON, PER CENT | THERMAL CONDUCTIVITY, *k*, CAL. PER CM. PER SEC. | | | |
|---|---|---|---|---|---|
| | | AT 100 C. (212 F.) | AT 200 C. (392 F.) | AT 300 C. (572 F.) | AT 400 C. (752 F.) |
| As cast | 0.68 | 0.122 | 0.117 | 0.113 | 0.107 |
| Annealed 40 hr | 0.12 | 0.136 | 0.128 | 0.122 | 0.115 |
| Annealed 80 hr | 0.11 | 0.134 | 0.127 | 0.122 | 0.116 |
| Annealed 120 hr | 0.09 | 0.134 | 0.126 | 0.120 | 0.114 |
| Annealed 160 hr | 0.12 | 0.130 | 0.124 | 0.118 | 0.113 |
| Annealed 200 hr | 0.12 | 0.132 | 0.124 | 0.117 | 0.112 |

The effect of annealing iron S8 is shown in Table XIV. All the irons showed the same effect of annealing as did the tests on ingot molds after use. The amount of increase depends on the degree of decomposition of the pearlite.

Donaldson states:

1. The thermal conductivity of gray cast iron varies from 0.110 to 0.137 cal. per cm. per sec., and decreases as the temperature increases.

2. The values obtained for thermal conductivity depend not only on composition but also, although to a much smaller extent, on structure.

3. The influence of silicon is to lower the conductivity considerably. Nickel has a similar effect, as have also manganese and vanadium, although to a lesser degree. Chromium and tungsten tend to raise the thermal conductivity.

4. As regards structure, free ferrite appears to be a much better conductor of heat than eutectoid pearlite.

5. Heat treatment at 550 C. (1020 F.) at first increases the thermal conductivity due to the pearlite decomposing to form ferrite and graphite, after which the oxidation of iron in the vicinity of the graphite flakes causes a slight decrease.

*Electrical Resistivity:*

Partridge(18) gives, among many others, the following values:

| | CHEMICAL ANALYSIS AS CAST | | | | SPECIFIC RESISTANCE, MICROHMS PER CU. CM. | |
|---|---|---|---|---|---|---|
| IRON | TOTAL CARBON, PER CENT | GRAPHITE, PER CENT | SILICON, PER CENT | MANGANESE, PER CENT | AS CAST | ANNEALED |
| No. 56......... | 3.66 | 2.00 | 1.33 | 0.02 | 78 | .... |
| No. 11......... | 3.06 | 1.71 | 2.54 | 0.02 | 86 | 75 |
| No. 12......... | 2.97 | 1.72 | 2.87 | 0.03 | 90 | 79 |
| No. 13......... | 2.97 | 1.80 | 3.41 | 0.03 | 108 | 99 |
| No. 14......... | 2.76 | 1.88 | 4.76 | 0.05 | 188 | 131 |
| No. 15......... | 2.61 | 2.52 | 6.04 | 0.09 | 192 | 191 |
| No. 16......... | 2.40 | 2.30 | 7.38 | 0.10 | 218 | 214 |

Manganese up to 2.66 per cent showed a very slight increase. Resistivity rose from 50 at 0.87 per cent aluminum to 95 at 4.09 per cent aluminum. It fell from 83 at 1.86 per cent cobalt to 34 at 23.3 per cent cobalt. Some results with increasing nickel, given below, are interesting:

| NICKEL, PER CENT | SPECIFIC RESISTANCE, MICROHMS PER CU. CM. | NICKEL, PER CENT | SPECIFIC RESISTANCE, MICROHMS PER CU. CM. |
|---|---|---|---|
| 0.71.............. | 44 | 14.50.............. | 122 |
| 2.81.............. | 48 | 16.45.............. | 114 |
| 5.40.............. | 81 | 14.83.............. | 119 |
| 8.86.............. | 110 | 16.48.............. | 127 |
| 11.51.............. | 115 | 18.83.............. | 134 |

*Magnetic Properties:*

Hatfield(19) says: "As in the case of electrical resistivity, the magnetic properties of cast iron may vary between very wide limits, ranging from those of materials having low permeability and high coercive force suitable for permanent magnets, and those having high permeability, low coercive force and low hysteresis loss suitable for electrical machinery."

Partridge(18) has made a comprehensive investigation of the magnetic properties and some of his results are shown in Tables XV and XVI.

TABLE XV.—HYSTERESIS CHARACTERISTICS (PARTRIDGE).

| | No. 11 | | No. 12 | | No. 13 | | No. 14 | | No. 15 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | As Cast | Annealed | As Cast | Annealed | As Cast | Annealed | As Cast | Annealed | As Cast | As Cast | Annealed |
| Limits of induction..... | 10 000 | 9 000 | 10 000 | 9 000 | 10 000 | 9 000 | 10 000 | 9 000 | 8 000 | 4 500 | 8 000 |
| Coercive force.. | 12.7 | 4.6 | 13.1 | 4.4 | 12.0 | 4.4 | 9.8 | 3.1 | 2.2 | 1.7 | 2.1 |
| Hysteresis loss, watts per lb.. | 20.0 | .... | 20.2 | .... | 18.0 | .... | 14.5 | .... | 2.7 | 1.6 | .... |

TABLE XVI.—NORMAL INDUCTION (Partridge).

| | No. 11 | | No. 12 | | No. 13 | | No. 14 | | No. 15 | | No. 16, As Cast |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | As Cast | Annealed | As Cast | Annealed | As Cast | Annealed | As Cast | Annealed | As Cast | Annealed | |
| *Bmax* (magnetic flux density).. | 10 190 | 10 980 | 10 100 | 10 850 | 9845 | 10 350 | 9455 | 9950 | 9200 | 9125 | 8935 |
| *Hmax* (field strength)..... | 99.8 | 100 | 100.1 | 100 | 100.2 | 100 | 100.1 | 100 | 99.7 | 100 | 97.3 |
| *μmax* (permeability)....... | 230 | 680 | 218 | 667 | 213 | 758 | 193 | 795 | 1021 | 1020 | 383 |
| *H* for *μmax* .... | 16.4 | 5.0 | 16.4 | 5.0 | 15.2 | 4.0 | 20.5 | 2.6 | 2.3 | 1.8 | 5.9 |

Partridge(20) draws the following general conclusions:

"The highest magnetic induction and permeability are obtained with cast iron which has been annealed. . .

"Carbon has a very marked influence on the magnetic properties. If present as graphite in the nodular form, high induction is obtained; if in the form of flakes, the specimen possesses low magnetic induction. If the ground mass is ferrite, low hysteresis loss and high permeability appear to be fundamental properties of the material. If the specimen has a pearlite matrix, it also will possess high hysteresis loss and comparatively low permeability. . .

"Graphite does not affect the hysteresis loss, but prevents the attainment of very high magnetic induction. This is due to lack of magnetic continuity of the structure. This lack of continuity causes small demagnetizing forces to act within the specimen. . .

". . . Phosphorus does not appear to have any marked effect on the permeability or induction. . .

"The addition of nickel decreases the magnetic permeability of cast iron to such an extent that it becomes practically non-magnetic when about 15 per cent of this element is present. . . The following show some interesting results on adding nickel to a low-silicon iron (0.32 per cent to 0.16 per cent):

| | 0.71 per cent Nickel | 2.81 per cent Nickel | 5.40 per cent Nickel | 8.86 per cent Nickel | 11.51 per cent Nickel | 14.50 per cent Nickel | 16.45 per cent Nickel | 14.83 per cent Nickel | 16.48 per cent Nickel | 18.83 per cent Nickel |
|---|---|---|---|---|---|---|---|---|---|---|
| $B_{max}$ ($H = 120$).. | 8900 | 10 320 | 5110 | 900 | 440 | 350 | 147 | 164 | 145 | 157 |
| Permeability (max.) | 193.0 | 165.0 | 55.0 | 7.5 | 3.2 | 3.0 | 1.4 | 1.6 | 1.8 | 1.4 |
| Total carbon, per cent......... | 3.66 | 3.60 | 3.52 | 3.46 | 3.18 | 3.16 | 3.02 | 3.16 | 2.82 | 2.93 |
| Graphitic carbon, per cent......... | 0.52 | 1.56 | 1.78 | 2.02 | 1.99 | 1.98 | 2.07 | 2.04 | 1.83 | 2.05 |

"Cobalt increases the induction, remanent magnetization, and permeability when added to cast iron, the high induction being due to the graphite existing in a very fine state of division. The presence of this element also increases the density, but it has very little effect on the coercive force and hysteresis loss, both of which are high. Annealing increases the maximum permeability and decreases the hysteresis loss, coercive force, and remanent magnetism."

## MECHANICAL PROPERTIES

### *Tensile Strength:*

Tensile strength has been adopted in this country as a basis for a working classification. A.S.T.M. Tentative Specifications A 48 – 32 T[1] lists the following seven tensile classes of gray iron:

| CLASS | TENSILE STRENGTH, MINIMUM, LB. PER SQ. IN. |
|---|---|
| No. 20 | 20 000 |
| No. 25 | 25 000 |
| No. 30 | 30 000 |
| No. 35 | 35 000 |
| No. 40 | 40 000 |
| No. 50 | 50 000 |
| No. 60 | 60 000 |

---

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 625 (1932); also 1932 Book of A.S.T.M. Tentative Standards, p. 179.

In general, the overall cost of production usually will increase with increase in tensile strength; therefore it is not economical to demand a higher strength iron than is required for the purpose intended.

Considering tensile strength as a fundamental property of cast iron and one that is easily determined, it perhaps will be most serviceable at this time to show in a general way the relations which exist between the other strength properties and the tensile strength. However, it must be borne in mind that individual producers can, for special purposes, produce rather remarkable combinations of apparently inconsistent properties, especially by means of alloying or heat treatment. The section on General Influence of Mass and Thermal History on Structural Make-up[1] treats of the effect of mass, thermal history, etc., so in the following discussion it is understood that simple shapes of approximately similar cooling rates are compared.

### *Modulus of Rupture:*

The term "modulus of rupture" is a mathematical convention adopted for expressing the transverse breaking strength (flexural strength) of materials which are of low ductility and which, in general, depart from Hooke's law, which is, that "stress divided by strain is constant."

The general formula for mid-span loading is $MR = \frac{Plc}{4I}$

where $MR$ = modulus of rupture in pounds per square inch,
$P$ = breaking load in pounds,
$l$ = distance between supports in inches,
$c$ = distance from neutral axis to extreme fiber, and
$I$ = moment of inertia.

For the three test bars of A.S.T.M. Specifications A 48 - 32 T the numerical values of $\frac{lc}{4I}$ are as follows:

| | |
|---|---|
| Diameter of bar 0.875 in., broken on 12-in. supports, $\frac{lc}{4I}$....... | 45.61 |
| Diameter of bar 1.20 in., broken on 18-in. supports, $\frac{lc}{4I}$....... | 26.53 |
| Diameter of bar 2.00 in., broken on 24-in. supports, $\frac{lc}{4I}$....... | 7.64 |

It therefore is necessary only to multiply the corrected breaking load of the bar by the corresponding number to obtain the modulus of rupture.

---

[1] See p. 141.

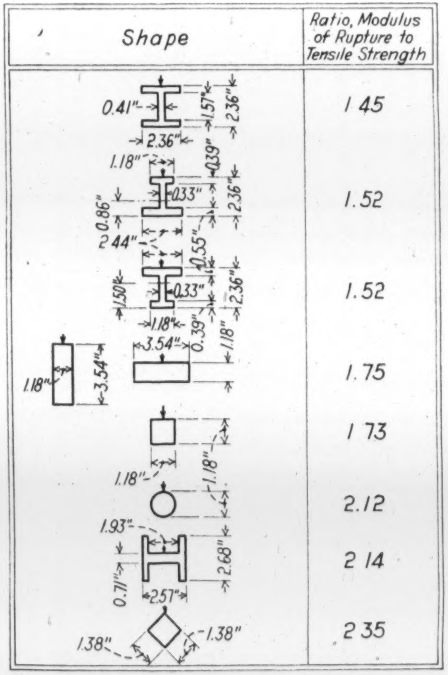
Shape
Ratio, Modulus of Rupture to Tensile Strength
0.41"
2.36"
1.57"
2.36"
1 45
1.18"
0.39"
0.33"
2.36"
0.86"
1.52
2 44"
0.55"
1.50"
0.33"
2.36"
1.52
1.18"
3.54"
0.39"
1.18"
1.18"
3.54"
1.75
1.18"
1.18"
1 73
2.12
1.93"
0.71"
2.68"
2.57"
2 14
1.38"
1.38"
2 35

FIG. 21.—Relation of Modulus of Rupture to Section. (Bach's "Elastizität und Festigkeit.")

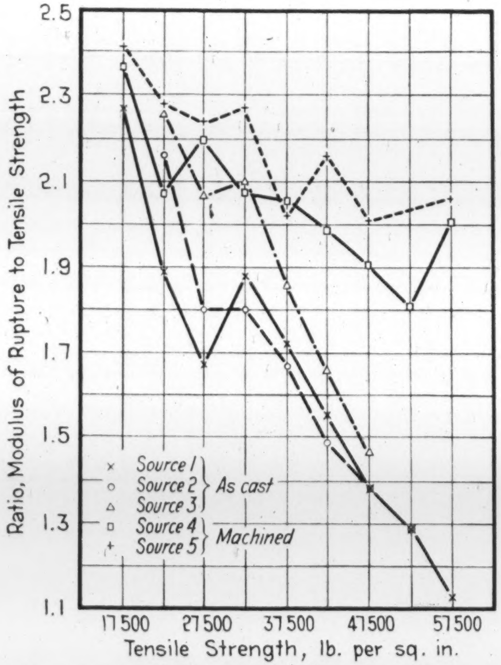

FIG. 22.—Relation of Tensile Strength to Ratio of Modulus of Rupture to Tensile Strength for Five Irons (Three as Cast, Two Machined).

TABLE XVII.—RELATION OF MODULUS OF RUPTURE TO TENSILE STRENGTH.

| TENSILE STRENGTH, LB. PER SQ. IN. | SOURCE[a] | NUMBER OF TESTS, $n$ | AVERAGE RATIO, $\bar{X}$ | ROOT MEAN SQUARE DEVIATION, $\sigma$ | MINIMUM RATIO | MAXIMUM RATIO |
|---|---|---|---|---|---|---|
| 15 100 to 20 000 | 1 | 8 | 2.27 | 0.51 | 1.77 | 3.11 |
| | 2 | 0 | .... | .... | .... | .... |
| | 3 | 0 | .... | .... | .... | .... |
| | 4 | 5 | 2.37 | 0.21 | 2.08 | 2.55 |
| | 5 | 5 | 2.41 | 0.26 | 2.09 | 2.87 |
| 20 100 to 25 000 | 1 | 22 | 1.89 | 0.34 | 1.52 | 2.58 |
| | 2 | 2 | 2.16 | .... | 2.03 | 2.28 |
| | 3 | 3 | 2.26 | 0.19 | 1.99 | 2.40 |
| | 4 | 3 | 2.07 | 0.30 | 1.65 | 2.35 |
| | 5 | 3 | 2.28 | 0.09 | 2.15 | 2.37 |
| 25 100 to 30 000 | 1 | 11 | 1.67 | 0.05 | 1.60 | 1.76 |
| | 2 | 4 | 1.80 | 0.14 | 1.57 | 1.94 |
| | 3 | 1 | 2.07 | .... | .... | .... |
| | 4 | 2 | 2.20 | .... | 2.14 | 2.26 |
| | 5 | 2 | 2.24 | .... | 2.15 | 2.32 |
| 30 100 to 35 000 | 1 | 19 | 1.88 | 0.16 | 1.48 | 2.11 |
| | 2 | 2 | 1.80 | .... | 1.66 | 1.93 |
| | 3 | 2 | 2.10 | .... | 2.02 | 2.17 |
| | 4 | 7 | 2.08 | 0.05 | 1.98 | 2.22 |
| | 5 | 7 | 2.27 | 0.15 | 2.11 | 2.54 |
| 35 100 to 40 000 | 1 | 10 | 1.72 | 0.16 | 1.38 | 2.11 |
| | 2 | 6 | 1.67 | 0.12 | 1.54 | 1.91 |
| | 3 | 5 | 1.86 | 0.17 | 1.67 | 2.14 |
| | 4 | 3 | 2.06 | 0.03 | 2.03 | 2.10 |
| | 5 | 2 | 2.02 | .... | 2.01 | 2.02 |
| 40 100 to 45 000 | 1 | 14 | 1.55 | 0.13 | 1.35 | 1.72 |
| | 2 | 7 | 1.49 | 0.13 | 1.32 | 1.68 |
| | 3 | 2 | 1.66 | .... | 1.47 | 1.85 |
| | 4 | 2 | 1.99 | .... | 1.96 | 2.02 |
| | 5 | 2 | 2.16 | .... | 2.12 | 2.19 |
| 45 100 to 50 000 | 1 | 7 | 1.38 | 0.10 | 1.26 | 1.56 |
| | 2 | 4 | 1.38 | 0.03 | 1.35 | 1.42 |
| | 3 | 3 | 1.47 | 0.09 | 1.40 | 1.59 |
| | 4 | 1 | 1.91 | .... | .... | .... |
| | 5 | 1 | 2.01 | .... | .... | .... |
| 50 100 to 55 000 | 1 | 4 | 1.29 | 0.12 | 1.21 | 1.50 |
| | 2 | 2 | 1.29 | .... | 1.21 | 1.36 |
| | 3 | .. | .... | .... | .... | .... |
| | 4 | 1 | 1.81 | .... | .... | .... |
| | 5 | 1 | 2.03 | .... | .... | .... |

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[a] Source 1—Plain cast iron in ten years American literature.
Source 2—MacKenzie(22).
Source 3—Thum and Ude(23).
Source 4—A-3 impact investigation(2) (machined) $\frac{\text{1.20-in. transverse bar}}{\text{0.8-in. tension bar}}$.
Source 5—A-3 impact investigation(2) $\frac{0.5 \times 5\text{-in. transverse bar}}{\text{0.5-in. tension bar}}$.

TABLE XVII.—*Continued.*

| TENSILE STRENGTH, LB. PER SQ. IN. | SOURCE[a] | NUMBER OF TESTS, $n$ | AVERAGE RATIO, $\overline{X}$ | ROOT MEAN SQUARE DEVIATION, $\sigma$ | MINIMUM RATIO | MAXIMUM RATIO |
|---|---|---|---|---|---|---|
| 55 100 | 1 | 1 | 1.13 | .... | .... | .... |
| | 2 | .. | .... | .... | .... | .... |
| | 3 | .. | .... | .... | .... | .... |
| | 4 | 1 | 2.01 | .... | .... | .... |
| | 5 | 1 | 2.06 | .... | .... | .... |

[a] See footnote, p. 58.

The modulus of rupture, strictly speaking, is not a property of the material, but varies with the shape[1] of the specimen, the conditions of loading, the surface conditions of the transverse bar (the tension bar being considered machined), and the degree of divergence from Hooke's law.

---

[1] The variation of modulus of rupture with the shape of section is shown in Fig. 21 taken from Bach(**21**), "Elastizität und Festigkeit." As corroborative evidence the difference in the modulus of rupture of the 1.20 by 18-in. bar (round) and that of the 2 by 1 by 24-in. bar (pipe) (rectangular) as reported (**22**) to the A.S.T.M. is as follows:

$$\text{Ratio modulus of rupture } \frac{\text{2 by 1 by 24-in. bar}}{\text{1.20 by 18-in. bar}}$$

| | |
|---|---|
| For 39 sets of bars (3 bars in each set) average ratio, $\overline{X}$ | 0.84 |
| Root mean square deviation, $\sigma$ | 0.065 |
| Absolute minimum ratio | 0.68 |
| Absolute maximum ratio | 1.02 |

The value for rectangular divided by round from Bach in Fig. 21 is 0.83.

Another valuable comparison is available from the Report on Impact Investigation(2) to be presented this year, where is given the results on the rectangular Frémont specimen 8 by 10 by 30 mm. and a test bar which was 0.5 in. in diameter tested on 5-in. supports. The data are as follows:

| | |
|---|---|
| For 24 sets of bars (3 bars in each set) average ratio, $\overline{X}$ | 0.83 |
| Root mean square deviation, $\sigma$ | 0.05 |
| Minimum ratio | 0.73 |
| Maximum ratio | 0.90 |

The effect of span is comparatively small within the limits usually tested. Meyersberg gives the following:

| RATIO OF SPAN TO DIAMETER | MODULUS OF RUPTURE |
|---|---|
| 20 | 100 |
| 15 | 104 |
| 11 | 100 |

MacKenzie (in an unpublished manuscript) shows the following ratios for the average of irons of A.S.T.M. Specifications A 48 – 32 T classes 20, 25 and 30 on bars 2.2 in. in diameter which reveals a continuous change in modulus of rupture but a critical point for relative modulus of elasticity at something below eight diameters. The results for the several classes differed very little from the average.

| SPAN, IN. | SPAN, DIAMETERS | PERCENTAGE OF AVERAGE VALUES FOR ALL SPANS | |
|---|---|---|---|
| | | MODULUS OF RUPTURE | MODULUS OF ELASTICITY |
| 48 | 21.8 | 93.5 | 111.7 |
| 36 | 16.4 | 96.0 | 111.4 |
| 24 | 10.8 | 99.0 | 109.1 |
| 18 | 8.2 | 100.2 | 107.3 |
| 12 | 5.5 | 102.5 | 89.3 |
| 8 | 3.6 | 109.2 | 71.2 |

The variation of modulus of rupture with tensile strength is worthy of more extended treatment. In general, the factors which tend to lower the tensile strength, such as high graphite content or large grain size, to mention the most important ones, also tend to cause large variations from Hooke's law. Therefore the higher the tensile strength the nearer the modulus of rupture approached it and the lower the tensile strength the further does the modulus of rupture lie above it.

Where cast iron is used as a beam, as in scale parts, or as a ring, as in water and culvert pipe subjected to earth loads, the modulus of rupture should be used in design. The high ratio of modulus of rupture to tensile strength is quite a factor in the design of such parts. For instance, a water pipe which is designed for internal pressure at 15,000 lb. per sq. in. tensile strength may safely be used at 40,000 lb. per sq. in. modulus of rupture against earth and truck loads.

Also, the softer and more plastic the iron, the less effect is introduced by surface defects of the "as cast" transverse bar. Another reason for this less effect on soft irons is the fact that the centers of bars of these irons are likely to be weak in comparison with the edges on account of the slower cooling. This condition is not so common with the high-strength irons[1] where, as a rule, shrinkage is the only factor tending to reduce the tensile strength.

Table XVIII gives the corresponding data for the 2.20-in. bars. The ratios and their rate of change are very similar to the results in Table XVII.

The data given by Kommers(24) on the ratio for the modulus of rupture of the 1.25 by 12-in. bar to a tension specimen 0.6 in. in diameter show the same tendency as in Table XIX.

---

[1] Table XVII gives some statistical data on this difference of soft and strong irons from several sources. No. 1 is a summary of most of the original values published in the United States during the last ten years on plain cast irons. No. 2 was taken from the data reported by MacKenzie(22) and tensile results subsequently added by unpublished report to the Editorial Committee. No. 3 was taken from a paper by Thum and Ude(23) in *Die Giesserei*, 1926. No. 4 is the ratio between the 1.20 by 18-in. machined transverse bar and the 0.8-in. tension bar from the Impact Report(2). No. 5 is the ratio between the 0.5 by 5-in. transverse bar and the 0.5-in. tension bar, also from the Impact Report(2). These results are graphically shown in Fig. 22. The relation is striking enough to pass without comment, except to remark that one iron of source 4, class 20 (20,100 to 25,000 lb. per sq. in.) had, for its tensile strength, a very high Brinell hardness on the edge of the 1.20-in. bar (due to high phosphorus, 1.99 per cent) and so is out of position. The average of the other two sets in this class was 2.18, which takes the big dip out of this curve at 22,500 lb. per sq. in. tensile strength. Also the marked effect of machining in the strong irons and the fading out of the difference in the weaker irons.

TABLE XVIII.—RATIO OF MODULUS OF RUPTURE TO TENSILE STRENGTH, 2.20-IN. DIAMETER TRANSVERSE BARS TO 1.25-IN. TENSION BARS (MACKENZIE(22)).

| TENSILE STRENGTH, LB. PER SQ. IN. | NUMBER OF TESTS, $n$ | AVERAGE RATIO, $\bar{X}$ | ROOT MEAN SQUARE DEVIATION, $\sigma$ | MINIMUM RATIO | MAXIMUM RATIO |
|---|---|---|---|---|---|
| 13 400 | 2 | 2.45 | .... | 2.42 | 2.47 |
| 18 600 | 3 | 2.42 | 0.33 | 2.10 | 2.84 |
| 23 000 | 5 | 2.23 | 0.21 | 2.01 | 2.64 |
| 28 200 | 2 | 2.09 | .... | 2.03 | 2.15 |
| 32 600 | 6 | 1.96 | 0.14 | 1.82 | 2.13 |
| 36 600 | 3 | 1.85 | 0.04 | 1.81 | 1.91 |
| 41 200 | 3 | 1.67 | 0.07 | 1.60 | 1.76 |
| 47 200 | 1 | 1.41 | .... | .... | .... |

TABLE XIX.—RELATION OF MODULUS OF RUPTURE TO TENSILE STRENGTH.

| AVERAGE TENSILE STRENGTH, LB. PER SQ. IN. | NUMBER OF TESTS, $n$ | AVERAGE RATIO, $\bar{X}$ | ROOT MEAN SQUARE DEVIATION, $\sigma$ | MINIMUM RATIO | MAXIMUM RATIO |
|---|---|---|---|---|---|
| 24 900 | 1 | 2.17 | .... | .... | .... |
| 31 900 | 4 | 1.94 | 0.11 | 1.80 | 2.04 |
| 36 800 | 3 | 1.89 | 0.19 | 1.64 | 2.10 |
| 49 200 | 2 | 1.67 | .... | 1.50 | 1.83 |

TABLE XX.—RELATION BETWEEN COMPRESSIVE AND TENSILE STRENGTH.

Compressive strength 1.20 by 2.4-in. bar, tensile strength 0.8-in. diameter bar.

| Tensile Strength, lb. per sq. in. | Impact Report(2) | | | | | Kommers(24) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Number of Tests, $n$ | Average Ratio, $\bar{X}$ | Root Mean Square Deviation, $\sigma$ | Minimum Ratio | Maximum Ratio | Number of Tests, $N$ | Average Ratio, $\bar{X}$ | Root Mean Square Deviation, $\sigma$ | Minimum Ratio | Maximum Ratio |
| 15 000 to 20 000 | 4 | 4.08 | 0.30 | 3.59 | 4.35 | .. | .... | .... | .... | .... |
| 20 100 to 25 000 | 4 | 4.02 | 0.31 | 3.67 | 4.50 | 1 | 3.42 | .... | .... | .... |
| 25 100 to 30 000 | 2 | 3.68 | .... | 3.67 | 3.69 | .. | .... | .... | .... | .... |
| 30 100 to 35 000 | 7 | 3.61 | 0.24 | 3.37 | 4.21 | 4 | 3.83 | 0.14 | 3.64 | 4.03 |
| 35 100 to 40 000 | 3 | 3.39 | 0.11 | 3.23 | 3.49 | 3 | 3.43 | 0.25 | 3.10 | 3.71 |
| 40 100 to 45 000 | 2 | 2.99 | .... | 2.85 | 3.13 | .. | .... | .... | .... | .... |
| 45 100 to 50 000 | 1 | 3.31 | .... | .... | .... | 1 | 3.25 | .... | .... | .... |
| 50 100 to 55 000 | 1 | 3.08 | .... | .... | .... | 1 | 3.07 | .... | .... | .... |
| 55 100 to 60 000 | 1 | 2.45 | .... | .... | .... | .. | .... | .... | .... | .... |

TABLE XXI.—RELATION OF COMPRESSIVE STRENGTH TO TENSILE STRENGTH.

| TENSILE STRENGTH, LB. PER SQ. IN. | RATIO, COMPRESSIVE TO TENSILE STRENGTH |
|---|---|
| 30 000 | 4.28 |
| 31 000 | 4.31 |
| 34 100 | 3.77 |
| 41 500 | 3.40 |

*Compressive Strength:*

The compressive strength of cast iron is one of its most valuable commercial properties but references are scarce as compared to the number of tension and transverse tests reported. Perhaps the best available list is that given in the Impact Investigation(2), which shows that the ratio of compressive strength to tensile strength varies inversely as the tensile strength, similarly to the modulus of rupture. A statistical exhibit from this report is shown in Table XX and for comparison the results of Kommers(24).

TABLE XXII.—RELATION OF SHEAR TO TENSILE STRENGTH, 0.5-IN. BARS.

| Tensile Strength, lb. per sq. in. | Impact Report(2) | | | | | Bolton(26) |
|---|---|---|---|---|---|---|
| | Number of Tests, $n$ | Average Ratio, $\overline{X}$ | Root Mean Square Deviation, $\sigma$ | Minimum Ratio | Maximum Ratio | Average Ratio, $\overline{X}$ |
| 15 100 to 20 000 | 5 | 1.64 | 0.21 | 1.38 | 2.03 | 1.57 |
| 20 100 to 25 000 | 3 | 1.38 | 0.07 | 1.29 | 1.47 | 1.44 |
| 25 100 to 30 000 | 2 | 1.38 | .... | 1.33 | 1.42 | 1.36 |
| 30 100 to 35 000 | 7 | 1.40 | 0.06 | 1.34 | 1.52 | 1.31 |
| 35 100 to 40 000 | 3 | 1.32 | 0.07 | 1.24 | 1.37 | 1.23 |
| 40 100 to 45 000 | 2 | 1.15 | .... | 1.13 | 1.16 | 1.18 |
| 45 100 to 50 000 | 1 | 1.29 | .... | .... | .... | .... |
| 50 100 to 55 000 | 1 | 1.19 | .... | .... | .... | .... |
| 55 100 to 60 000 | 1 | 1.01 | .... | .... | .... | .... |

TABLE XXIII.—TORSION STRENGTH OF CAST IRON (BACH(27)).

Tensile Strength: Cast Iron A—22,500 lb. per sq. in.
Cast Iron B—23,900 lb. per sq. in.
Cast Iron C—32,000 lb. per sq. in.

| | RATIO, TORSIONAL STRENGTH TO TENSILE STRENGTH | | |
|---|---|---|---|
| | CAST IRON A | CAST IRON B | CAST IRON C |
| Round | 1.02 | .... | .... |
| Hollow ring | 0.82 | 0.86 | .... |
| Rectangle: | | | |
| Ratio, base to height = 1:1 | 1.42 | 1.55 | .... |
| = 1:25 | 1.60 | .... | 1.42 |
| = 1:5 | 1.50 | 1.59 | .... |
| = 1:9 | 1.59 | .... | .... |
| Hollow square | 1.13 | .... | .... |
| Trapezoid | .... | .... | 1.55 |
| Triangle equilateral | .... | .... | 1.52 |
| Triangle with one sharp angle | .... | .... | 1.38 |

Piwowarsky lists(25) very few compressive strengths—the examples of Table XXI.

Not knowing the method of test, it is difficult to comment on the absolute ratios which lie somewhat above those in Table XX, but the change with increasing tensile strength is about the same.

*Shear Strength:*

Shear strength has been given much publicity by the French testing engineers. It is an excellent test for the local strength of a casting and as such is especially useful in "exploring" a casting. Its relation to the tensile strength is quite similar to that of modulus of rupture and compression. The shear test results obtained in the

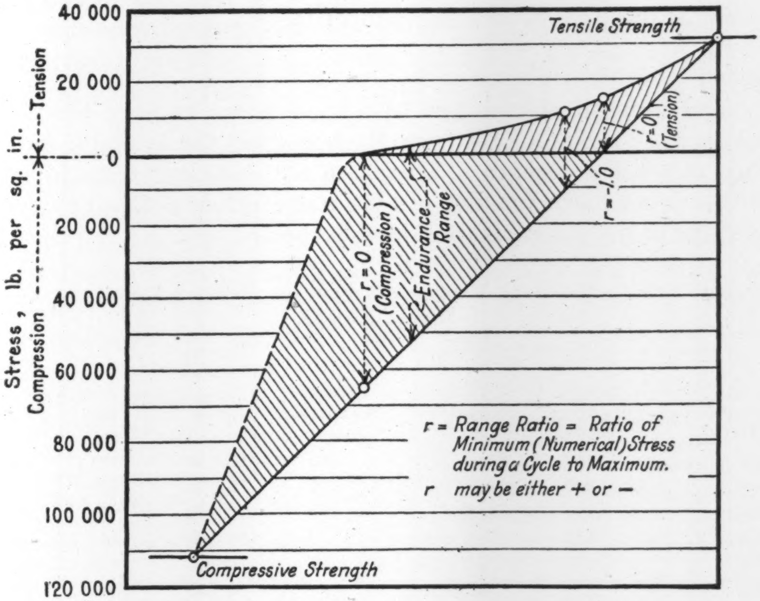

FIG. 23.—Diagram of Varying Endurance Limits of Cast Iron with Varying Range of Stress. (J. B. Kommers.)

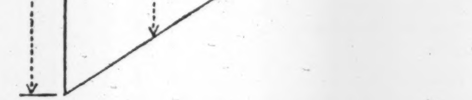

FIG. 24.—Portion of Diagram Shown in Fig. 21, from Maximum Compression Endurance to Maximum Tensile Endurance Range. (J. B. Kommers.)

Impact Investigation(2) check data given in a paper previously published by the American Foundrymen's Association(26). They are shown together in Table XXII. Marked variation in relationship of shear to tensile strength is encountered in irons of high phosphorus content or those containing free cementite.

### *Torsion:*

The strength of cast iron in torsion seems to have been little considered. Table XXIII from Bach(27) gives the available data in terms of tensile strength. There are not sufficient data to judge whether it is a linear function of tensile strength or not, but evidently the shape of the piece has considerable effect.

TABLE XXIV.—RELATIVE VALUES FOR SURFACE FATIGUE LIMIT AND LOAD STRESS FACTOR.

| Material in Pinion | Brinell Hardness Number | Material in Gear | Brinell Hardness Number | Surface Fatigue Limit, $S_f$, lb. per sq. in. | Load-Stress Factor, $K$ | |
|---|---|---|---|---|---|---|
| | | | | | 14½-deg. Gear | 20-deg. Gear |
| Steel........... | 150 | Steel......... | 150 | 50 000 | 30 | 41 |
| Steel........... | 200 | Steel......... | 200 | 70 000 | 58 | 79 |
| Steel........... | 250 | Steel......... | 250 | 90 000 | 96 | 131 |
| Steel........... | 150 | Cast iron..... | ... | 50 000 | 44 | 60 |
| Steel........... | 200 | Cast iron..... | ... | 70 000 | 87 | 119 |
| Steel........... | 250 | Cast iron..... | ... | 90 000 | 144 | 196 |
| Cast iron....... | ... | Cast iron..... | ... | 90 000 | 193 | 284 |

### *Endurance Limit:*

Recent investigations have shown that endurance limit or fatigue strength apparently varies linearly with the tensile strength. The statistics of the ratio, endurance limit to tensile strength, are given below:

| | Thum and Ude(23) | Kommers(24) | Impact Investigation(2) |
|---|---|---|---|
| Number of sets tested, $n$........... | 14 | 10 | 24 |
| Average ratio, $\overline{X}$.................. | 0.40 | 0.49 | 0.50 |
| Root mean square deviation, $\sigma$...... | 0.03 | 0.06 | 0.04 |
| Minimum ratio.................... | 0.35 | 0.42 | 0.43 |
| Maximum ratio.................... | 0.45 | 0.57 | 0.63 |

Kommers worked with machines carefully protected from vibration which probably accounts for his higher ratio, though some of the difference may be due to the small tension bar used for his comparison, whereas Thum and Ude used a standard tension bar (0.8 in.). The endurance specimens were 0.33 in. in diameter in Kommers' tests and 0.37 in. in Thum and Ude's. Moore showed(28) that for soft cast iron the effects of grooves and notches in the specimen is very much less than that expected for most other materials. A specimen of cast iron with a radial hole which should have reduced the endurance limit 67 per cent actually made a reduction of only 13 per cent. Two other specimens tested with filleted grooves which were expected to reduce the endurance limit 74 per cent actually showed 0 and 8 per cent reduction in the two cases. Kommers tested a soft cast iron with a square notch on the specimen and actually found an increase of 500 lb. per sq. in. Kommers shows[1] the interesting effect of reversed stress.

An important application of endurance properties is in the design of metal spur gears. The figures of Table XXIV (Proposed Recommended Practice of the American Gear Manufacturers' Association for Computing the Allowable Tooth Loads of Metal Spur Gears) show the relative values for surface fatigue limit and load stress factor for steel and cast iron of similar Brinell hardness in gears of 14½ and 20 deg.

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[1] J. B. Kommers, "The Fatigue Properties of Cast Iron," *Proceedings*, Am. Soc. Testing Mats. Vol. 29, Part II, pp. 103–107 (1929). "*Tests on Range of Stress:* Fig. 1 (accompanying Fig. 23) shows the results obtained when fatigue tests were made using various combinations of tension and compression in the cycle of applied stress. When the unit stress was entirely compressive from zero to a maximum ($r = 0$), the endurance range was 65,000 lb. per sq. in., representing the endurance limit in compression. When a small amount of tension was introduced into the cycle the endurance range was reduced at once. For example, at 6400 lb. per sq. in., tensile stress in the cycle the endurance range becomes 38,400 lb. per sq. in. For completely reversed stress, that is, tensile stress equal to compressive stress ($r = -1$), the endurance range is reduced to 21,000 lb. per sq. in., the endurance limit being 10,500 lb. per sq. in. Finally, when the unit stress was entirely tensile from zero to a maximum ($r = 0$), the endurance range was smallest of all, being only 15,500 lb. per sq. in., corresponding to the endurance limit in tension.

"The endurance range, therefore, in these tests varied from 65,000 lb. per sq. in. when the stress was entirely compressive, to 15,500 lb. per sq. in. when the stress was entirely tensile. It is evident, therefore, that for cast iron, tensile stress is especially effective in producing fatigue failure.

"If $S_{-1}$ is the endurance limit for completely reversed stress, $r$ is the ratio of the minimum to the maximum unit stress and $S_{max}$ is the maximum unit stress for any cycle, then the following equation may be used for that portion of Fig. 1 (accompanying Fig. 23) in which the tensile unit stress is numerically the maximum:

$$S_{max} = \frac{3\,S_{-1}}{2 - r} \quad \ldots\ldots (1)$$

For the portion of Fig. 1 (accompanying Fig. 23) in which the compressive stress is numerically the maximum $S_{max}$ may be obtained from the following equation:

$$S_{max} = \frac{6\,S_{-1}}{1 - 5r} \quad \ldots\ldots (2)$$

"Equation 2 covers the portion of the diagram shown in Fig. 2 (accompanying Fig. 24), which is the same as that of Fig. 1 (accompanying Fig. 23), in which the compressive unit stress is numerically the maximum, except that the upper limit of tensile stress has been approximated by a straight instead of a curved line. Taking the value of $S_{-1}$ as 10,500, the tests show that when $r = 0$ the endurance limit is practically $6\,S_{-1}$. When $r = -0.2$ the equation gives a value of 31,500 lb. per sq. in. for $S_{max}$. The experiments gave a value of 32,000 lb. per sq. in.

"It should be noted that in both equations 1 and 2, $r$ is minus when the cycle of stress is partly tensile and partly compressive."

*Elasticity, Plasticity, Stiffness:*

The term "modulus of elasticity," as applied to cast iron, generally means the relative stiffness of the iron under the particular conditions of loading. It may be determined directly in the tensile or compression test by measuring the change in length per unit of load. The formula for any given loading is:

$$E = \frac{Pl}{eA}$$

where $E$ = modulus of elasticity,
$P$ = load in pounds,
$l$ = gage length in inches,
$e$ = change of gage length in inches under load $P$, and
$A$ = cross-sectional area of specimen in square inches.

This is not the true tangential modulus for the given loading but furnishes a relative measure of rigidity.

The relative modulus of elasticity as determined in the transverse test is almost as simple a calculation as that in tension or compression. The general formula for a single centrally applied load is:

$$E = \frac{Pl^3}{48\,ID}$$

where $E$ = modulus of elasticity in pounds per square inch,
$P$ = load in pounds,
$l$ = span in inches,
$I$ = moment of inertia, and
$D$ = deflection (at load $P$) in inches.

The moment of inertia is:

$\frac{\pi d^4}{64}$ for a round bar, or $\frac{bd^3}{12}$ for a rectangular bar

where $b$ = breadth, and
$d$ = thickness or diameter.

For any standard bar the value $\frac{l^3}{48\,I}$ may be evaluated and used as a constant.

This, therefore, reduces to the following simple formulas for the three proposed standard bars:

For 0.875-in. diam. bar on 12-in. support, $E = 1251 \frac{P}{D}$

For 1.20-in. diam. bar on 18-in. support, $E = 1194 \frac{P}{D}$

For 2.00-in. diam. bar on 24-in. support, $E = 367 \frac{P}{D}$

Since the modulus varies rapidly with the dimensions, the load and deflection must be corrected to standard before these simple formulas can be used. The deflection may be converted to equivalent deflection of the standard size bar by the formula:

$$D = D_o \frac{d_o}{d_s}$$

where $D$ = corrected deflection at corrected load,
$D_o$ = observed deflection at actual load,
$d_o$ = measured diameter, and
$d_s$ = standard diameter.

For determining the relative modulus of elasticity at intermediate loads where the bar is over or under size, read the actual deflection at the desired load (corrected by the table in A.S.T.M. Specifications A 48 – 32 T) and correct the deflection at this point by the formula given above.

TABLE XXV.—RELATIONS BETWEEN MODULI IN COMPRESSION, TENSION, AND TRANSVERSE TESTS.

Number of sets tested = 25.

| | RATIO, RELATIVE MODULUS OF ELASTICITY IN TENSION TO THAT IN TRANSVERSE TESTS | RATIO, RELATIVE MODULUS OF ELASTICITY IN COMPRESSION TO THAT IN TRANSVERSE TESTS |
|---|---|---|
| Average ratio at $\frac{1}{4}$ breaking stress, $\overline{X}$ | 0.89 | 0.88 |
| Root mean square deviation, $\sigma$ | 0.05 | 0.10 |
| Minimum ratio | 0.75 | 0.69 |
| Maximum ratio | 0.97 | 1.02 |

The modulus of rigidity is the ratio of stress to strain in the shear and torsion tests. Actual determinations are very rare but the value 0.4 $E$ for the modulus of rigidity may be assumed within normal working limits if $E$ has been determined at about the same proportion of working stress to ultimate stress.

The relative modulus of elasticity in the transverse test is affected more by the length of span than by changes in shape. On the 39 sets of bars quoted under modulus of rupture, the following statistical relations between the relative modulus of elasticity of the 2 by 1 by 24-in. and the 1.20 by 18-in. bars were found:

| | |
|---|---|
| Average ratio, 2 by 1 by 24-in. bar to 1.20 by 18-in. bar, $\overline{X}$ | 0.99 |
| Root mean square deviation, $\sigma$ | 0.06 |
| Minimum ratio | 0.88 |
| Maximum ratio | 1.16 |

The ratio of span to moment of inertia is fairly close (144 and 120) on these two bars.

In the Impact Investigation(2) careful stress-strain curves were obtained up to 50 per cent in compression, 75 per cent in tension, and to 100 per cent in flexure, and the moduli were taken at 25 per cent of the ultimate in each case, about the limit of safe working stress, the statistical relations are found for the three tests, as given in Table XXV.

The relative modulus of elasticity in general increases as the tensile strength. The relation between these two properties is shown below from the Impact Investigation(2):

| Tensile Strength, lb. per sq. in. | Number of Tests, $n$ | Modulus of Elasticity, Average, lb. per sq. in., $\bar{X}$ | Root Mean Square Deviation, $\sigma$ | Modulus of Elasticity, lb. per sq. in. | |
|---|---|---|---|---|---|
| | | | | Minimum | Maximum |
| 15 100 to 20 000.......... | 4 | 11 600 000 | 0.9 | 10 400 000 | 12 800 000 |
| 20 100 to 25 000.......... | 4 | 14 200 000 | 0.5 | 13 300 000 | 14 600 000 |
| 25 100 to 30 000.......... | 2 | 14 500 000 | .... | 14 300 000 | 14 600 000 |
| 30 100 to 35 000.......... | 7 | 16 000 000 | 0.6 | 13 200 000 | 17 400 000 |
| 35 100 to 40 000.......... | 3 | 18 100 000 | 1.4 | 16 500 000 | 19 900 000 |
| 40 100 to 45 000.......... | 2 | 18 500 000 | .... | 17 900 000 | 19 100 000 |
| 45 100 to 50 000.......... | 1 | 22 600 000 | .... | ........ | ........ |
| 50 100 to 55 000.......... | 1 | 19 100 000 | .... | ........ | ........ |
| 55 100 to 60 000.......... | .. | 19 900 000 | .... | ........ | ........ |

The modulus is given at one-fourth the load in Table XXVI, since this is the most useful range in engineering practice. The values for relative moduli at ultimate load vary much more than those at the quarter load and are much lower.

Cast iron, when first subjected to stress, shows a distinct permanent deformation. A second repetition of the same stress gives very little more and after some five to ten loadings the metal behaves as a perfectly elastic material—a good high tensile strength iron showing no further plastic deformation after many hundreds of repetitions at 80 per cent of the breaking load. If, however, the material which has become completely elastic at one loading is subjected to a higher loading, it behaves as virgin material between the first load and the second. A bar which has been stressed several times to a certain load will give a "knee" on the curve if carried beyond this point. Also the metal will take an additional "set" if the temperature is raised.

The curves in Figs. 25 and 26 from Sugimura(29) show clearly this behavior, Fig. 25 showing 12 consecutive loadings of one bar at room temperature and Fig. 26 four loadings at increasing temperatures on the same bar.

The amount of "set" seems to be in general inversely proportional to the strength and modulus of elasticity. Figure 27 shows some relationships on five sets of transverse bars which illustrates the point. This set was at 80 per cent of the ultimate strength and

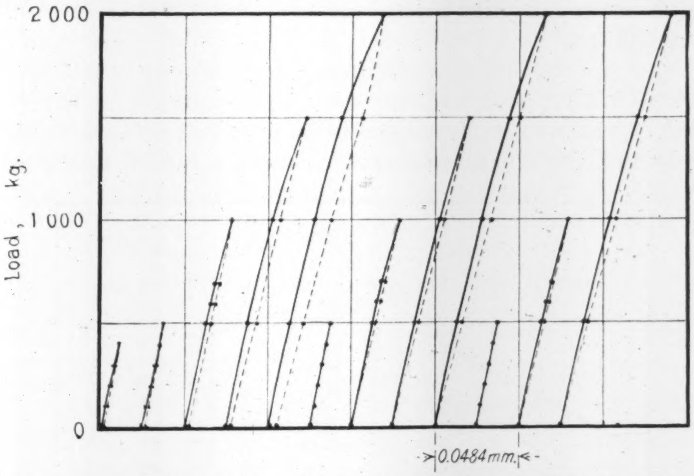

FIG. 25.—Curves Showing Results of Repeated Loading on Specimen in Tension. (I. Sugimura.)

Gage length, 7.6 cm. Diameter, 1.904 cm.

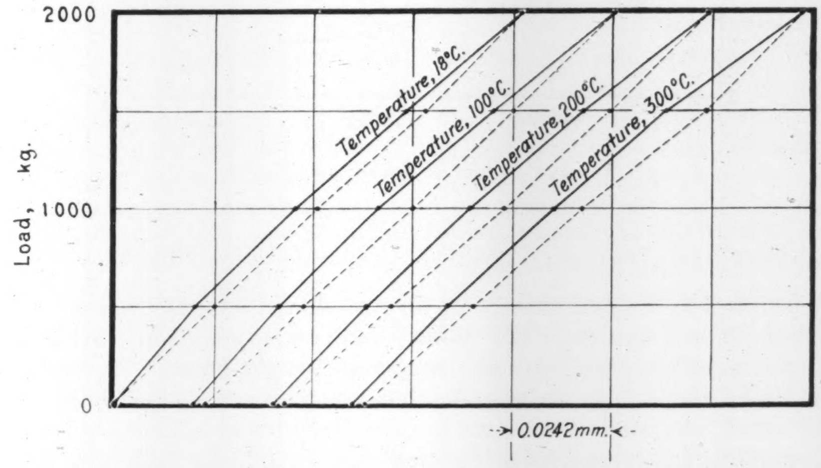

FIG. 26.—Curves Showing Results on Tests on Specimens Used in Fig. 23 at Ordinary and at Elevated Temperatures. (I. Sugimura.)

Gage length, 12.7 cm.

is consequently far more than would occur at reasonable loads for, as has been shown(30) the set at 80 per cent is over three times that at 25 per cent of the ultimate strength.

*Impact or Shock Resistance:*

Cast iron is not commonly used to resist impact or shock, yet as between different cast irons there are great differences in impact strength—some varieties being able to withstand considerable shock without failure.

*Types of Tests.*—The usual impact tests are of two general kinds. First, and most commonly used on tough materials, is the "swing-through" pendulum delivering a blow in excess of that required to cause failure, the energy absorbed in rupture of the specimen being registered as loss of energy of the pendulum—Izod, Charpy and direct impact tension. The second, and one long used for cast iron, is the repeated application of comparatively light blows. One modification, the Krupp-Stanton, is that of continued dropping of the hammer from a small constant height—the number of blows to break being the value reported. Another modification is that of a constantly increasing height of drop—the height of the last, or breaking, fall in inches being reported as the resistance of the material.

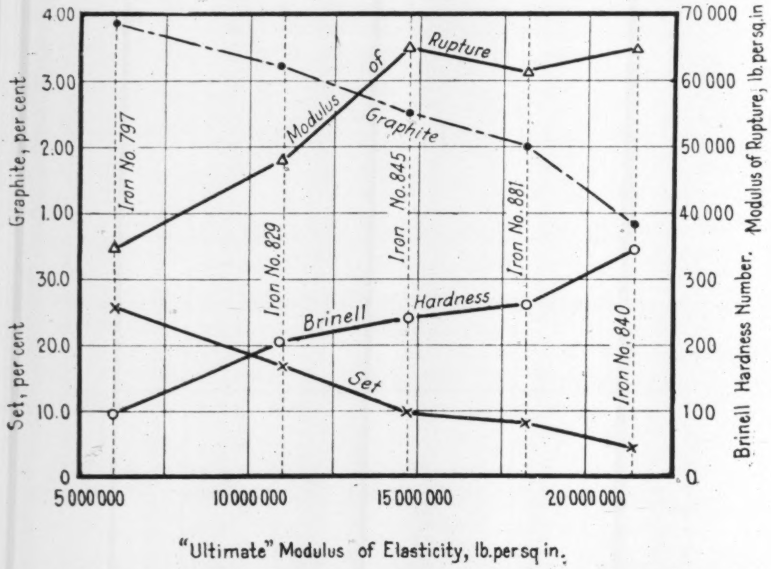

FIG. 27.—Values for Plastic Deformation (Set) in Six Repeated Loadings to Three-Fourths Ultimate Strength Plotted Against Ultimate Modulus of Elasticity, with Trend of Strength, Hardness and Graphite Indicated. (MacKenzie.)

*Variations in Results from Different Test Methods.*—It is clear from the conditions of the tests cited above that one material might be excellent in one test and not very good in another. For example, plastic deformation would cause considerable energy absorption in the single blow pendulum tests but would be of rather small moment in the increasing height hammer tests. Also, in the Krupp-Stanton type of test, the relative value of the endurance limit and the force of the blow would be of great importance while this would be a small factor indeed in the Charpy or Izod tests.

*Methods of Expression of Results.*—For materials which show the intercrystalline fracture under both slow and sudden application of load, the resilience or work done in breaking the specimen in the ordinary test—either tension, compression or transverse—should be a fairly good measure of the resistance of the material to a shock of the same nature, that is, tensile, compressive or transverse. This resilience is also defined as the area under the stress-strain curve, the "true" resilience. It may be approximated as $\frac{\text{load} \times \text{deflection}}{2}$ which is called the "triangular" resilience. The triangular resilience may also be expressed for any one set of conditions—span, approximate dimension, application of load, etc.—as the square of the modulus of rupture divided by the ultimate modulus of elasticity. The ratio of "true" to triangular resilience is in general an inverse function of the stiffness but an even better estimate can be gotten from the ratio of the relative elastic moduli at $\frac{1}{2}$ load and at breaking.

Committee A-3 on Cast Iron of the A.S.T.M. has conducted an investigation on impact testing of cast iron, this investigation being under the direction of J. T. MacKenzie, Chairman of Subcommittee XV on Impact Testing(2). The investigation covers 25 cast irons and includes a large range of compositions and melting methods. The report shows the results of various static and shock tests.

*Impact Tests and Resilience.*—A comparison between the several forms of pendulum impact tests and the resilience is shown in Fig. 28 which gives the comparison on bars of 1.20 in. in diameter and 18-in. span except one test which was run on an 8-in. span to see whether the smaller bars could be used for the impact test. The data are taken from the impact testing investigation referred to above. The curves are good except for $N$, a martensitic iron, and $X$, an austenitic iron, where the 8-in. span gives a value far above the 18-in. span. The 8-in. span lies above the other two curves as would be expected from the lower modulus of elasticity due to short span.

In the A.S.T.M. impact investigation, tests were made with notched and unnotched Charpy test bars (0.394 in. square), notched Izod bars (0.41 in. square) and unnotched Izod bars ($\frac{3}{4}$ by $\frac{1}{2}$ in.). These small bars gave results of questionable value and are not

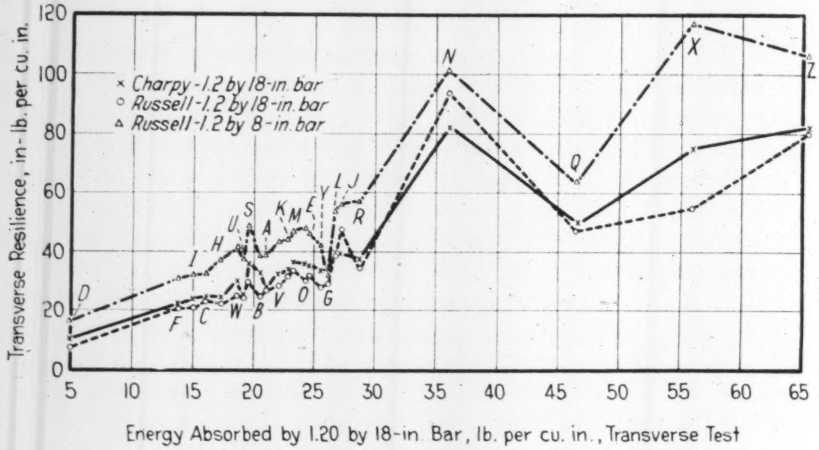

FIG. 28.—Relation of Pendulum Impact Value to Transverse Resilience (1.20-in. Diameter Bars).

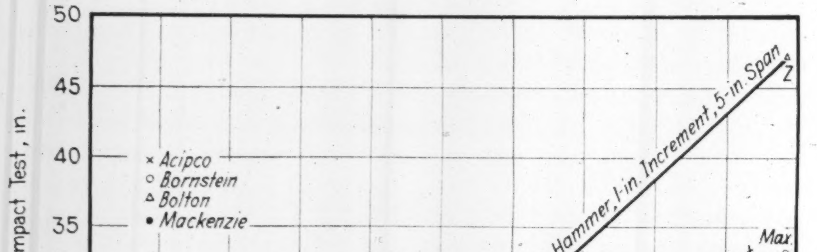

FIG. 29.—Results of Falling Hammer Test on 1.20 by 18-in. Bars Using Increasing Height of Drop.

recommended for impact testing of cast iron. Tests were made on larger (1.20-in. diameter) test bars (unnotched) in both Charpy and Russell testing machines. These gave much better results.

The results from the Krupp-Stanton test (4.8-lb. hammer, 1.2-in. drop, ½-in. diameter groove bar rotated 180 deg. after each blow) were plotted against transverse resilience and the curves showed a lack of relationship. This test cannot be recommended.

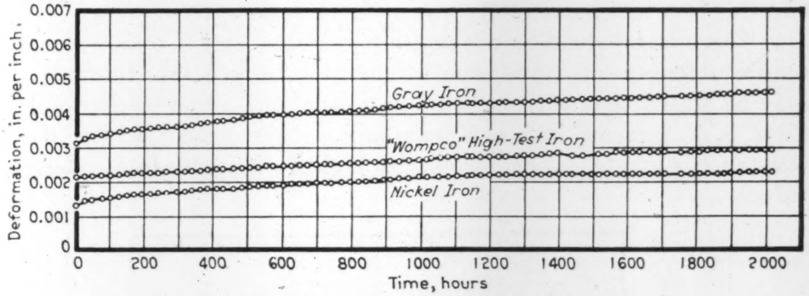

FIG. 30.—Time-Deformation Curves for Three Irons.

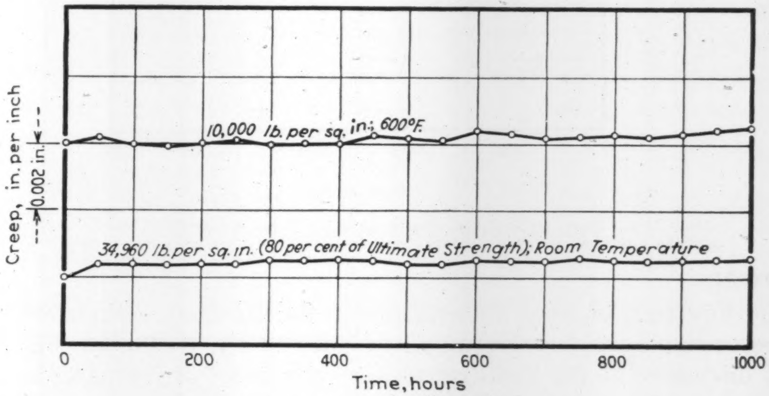

FIG. 31.—Creep-Time Curves.

The results of direct tension impact tests on ½-in. specimens were plotted against resilience, tensile strength, etc., and no logical relationship could be established. This test cannot be recommended.

The results of the falling hammer test using increasing height of drop are shown in Fig. 29. The bottom scale is an attempt to arrive at the resilience remaining after repeated stressing just below the breaking point. The machines used were rather crude but the tests seemed to show a very definite relation between these two properties. At present we are not able to translate the results of one particular machine in the terms of another testing machine. However, this test is a simple one and appears promising.

It is generally recognized that increasing phosphorus content reduces the impact or shock value of cast iron. In the A.S.T.M. impact investigation no attempt was made to determine the effect of phosphorus on impact resistance. However, one of the irons tested had an extremely high phosphorus content, 2.00 per cent, and this iron was particularly low in shock resistance.

There is no definite relationship between shock resistance and tensile strength or transverse strength. In general, however, it may be stated that with increase in tensile and transverse strengths, there is an increase in shock resistance. Also, generally, the shock resistance is greater for the irons with higher endurance limits (fatigue value).

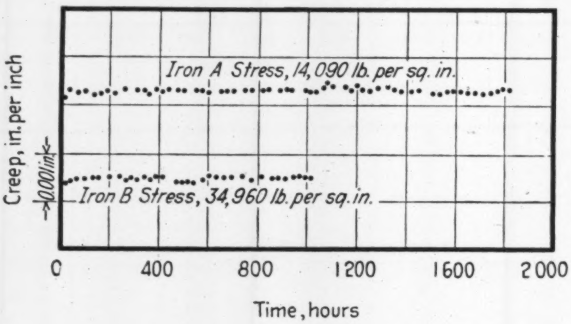

FIG. 32.—Creep Tests on Cast Iron at 100 F.

*Creep:*

Few tests of creep are available in the literature. Probably the most reliable are those given by R. J. Allen and J. W. Bolton (Fig. 30) in discussion of the high-temperature properties of cast iron in the Symposium on Effect of Temperature on Metals(31) (since more fully covered by Gregg(32)). This material is summarized in Table XXVI. Bolton's results (Fig. 31) on a plain cast iron of 45,000 lb. per sq. in. 2.72 per cent total carbon, show very little creep during 1000 hr. at load of 10,000 lb. per sq. in. at 600 F. (315 C.) (0.0025 in. per inch) and a similar test at 35,000 lb. per sq. in. stress (Fig. 32) at room temperature showed only 0.005 in., practically all of which occurred during the first two days. Bolton suggests that, in view of these data, the higher strength classes of cast iron may be useful considerably above the temperatures which the engineers are accustomed to consider for cast iron. The results shown in Fig. 32 are significant in that they show that a very low strength iron (tensile strength 17,610 lb. per sq. in.) can be loaded indefinitely at 14,090 lb. per sq. in. (80 per cent of its tensile strength) without appreciable creep.

*Hardness:*

Hardness is a result of a number of variables and cannot always be related to a single property. In general, and speaking largely of ordinary foundry irons, the indentation hardness such as Brinell and Rockwell increase as the tensile strength.

For following the daily production of one certain foundry, or for checking the quality of important castings, the Brinell or Rockwell hardness has real value. It should be used with great caution in specifications as it is not a simple function of any property whether it be strength, wear, machineability, or what not.

TABLE XXVI.—CREEP TESTS AT BATTELLE MEMORIAL INSTITUTE, 700 F.

| Total Carbon, per cent | Graphitic Carbon, per cent | Silicon, per cent | Alloy,[a] per cent | Tensile Strength, lb. per sq. in. | Brinell Hardness Number | End Limit, lb. per sq. in. | Stress, lb. per sq. in. | Deformation, per inch per hour × 10 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | 0 to 150 hr. | 150 to 450 hr. | 450 to 900 hr. | 900 to 1200 hr. | 1200 to 2000 hr. |
| 3.40 | 2.70 | 1.50 | none | 31 500 | 197 | 14 500 | 10 500 | 12.1 | 12.1 | 5.9 | 3.4 | 3.0 |
| 2.95 | 2.10 | 2.45 | none | 45 000 | 237 | 22 500 | 10 500 | 5.3 | 5.3 | 5.3 | 3.4 | 0.0 |
| 3.20 | 2.60 | 1.15 | Nickel-1.50 | 44 500 | 230 | 17 500 | 10 500 | .... | 8.2[b] | 5.3 | 0.0 | 0.0 |
| 2.75 | 2.27 | 2.10 | Molybdenum-0.83 | 59 500 | 241 | ...... | 10 500 | 0.8 | 0.8 | 2.2 | 0.0 | 0.0 |
| 2.72 | 2.06 | 2.50 | Molybdenum-0.83 | 52 500 | 241 | ...... | 17 300 | 10.0 | 0.0 | 0.0 | 0.0 | .... |
| 2.72 | 2.06 | 2.50 | Molybdenum-0.83 | 52 500 | 241 | ...... | 19 500 | 16.7 | 3.7 | 0.0 | 0.0 | .... |
| 2.72 | 2.06 | 2.50 | Molybdenum-0.83 | 52 500 | 241 | ...... | 24 000 | 30.0 | 6.7 | 1.6 | 0.0 | .... |

[a] Manganese about 0.7, phosphorus less than 0.2, sulfur less than 0.10, except the nickel iron which was 0.15.
[b] 0 to 500 hr.

The Brinell hardness test usually is applied as 3000-kg. load on a 10-mm. ball for 30 seconds, though there is practically no change in most irons after two or three seconds. For small specimens the 5-mm. ball may be used with 750-kg. load (thus keeping the stress per unit of area the same) and consequently the results check quite well.

The Rockwell hardness, "B" scale ($\frac{1}{16}$-in. ball), is commonly used for cast iron though some engineers use the diamond for hard irons like dies, rolls, etc., or the $\frac{1}{8}$-in. ball may be used for very soft specimens. The other hardness testers are also used with good results though with all the small penetrators care must be exercised to make a number of impressions if the iron is found to be open-grained so that the soft spots may be "drowned out." There is the danger, for instance, of reading low if the penetrator gets on a graphite flake or high if it gets on a phosphide island.

For chilled or white irons the scleroscope has been found to give good results.

Petrenko(33) investigated the Rockwell-Brinell relation and developed the following equation for converting from Rockwell hardness, "B" scale, to Brinell hardness:

$$\mathrm{BHN} = \frac{7300}{130 - R_{B_{\frac{1}{16}}}}$$

For the cast irons reported in the Impact Investigation(2) the equation found, by least squares, was:

$$\mathrm{BHN} = \frac{8030}{132 - R_{B_{\frac{1}{16}}}}$$

The difference is never over ten points Brinell or five points Rockwell in the useful range (70 to 105 Rockwell or 130 to 280 Brinell hardness number).

*Effect of Section Size:*

As is shown in the section on General Influence of Mass and Thermal History on Structural Make-up,[1] the properties of a specimen of cast iron are fundamentally a function of the cooling rate. In ordinary foundry practice, this means that the thickness of the section controls the cooling rate since all other factors are usually fairly constant.

The variation in strength with section or cooling rate has been studied by a number of investigators. The data given by Rother and Mazurie(34) are illustrative of a great number. These data are summarized below:

| | PERCENTAGE OF TENSILE STRENGTH OF 1.0-IN. DIAMETER BAR | | | |
|---|---|---|---|---|
| TENSILE STRENGTH, 1-IN. DIAMETER BAR, AS CAST, LB. PER SQ. IN. | 1½-IN. DIAMETER BAR | 2-IN. DIAMETER BAR | 2½-IN. DIAMETER BAR | 3-IN. DIAMETER BAR |
| 21 300 | 77 | 72 | 71 | 67 |
| 23 800 | 81 | 69 | 61 | 53 |
| 25 900 | 89 | 74 | 55 | 46 |
| ——— | — | — | — | — |
| Avg. 23 700 (soft iron) | 82 | 72 | 62 | 55 |
| 32 800 | 94 | 84 | 75 | 73 |
| 36 300 | 85 | 83 | 76 | 76 |
| ——— | — | — | — | — |
| Avg. 34 600 (machinery iron) | 90 | 84 | 76 | 75 |

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[1] See p. 141.

Bornstein(35) lately reported some carefully conducted tests as follows:

| Tensile Strength, 1.2-in. Diameter Bar, as Cast, lb. per sq. in. | Percentage of Tensile Strength of 1.2-in. Diameter Bar | | |
|---|---|---|---|
| | $\frac{3}{4}$-in. Diameter Bar | 2-in. Diameter Bar | 3-in. Diameter Bar |
| 23 600 | 122 | 65 | 51 |
| 25 400 | 107 | 71 | 57 |
| Avg. 24 500 (soft iron) | 115 | 68 | 54 |
| 35 200 | 104 | 69 | 53 |
| 36 000 | 104 | 77 | 63 |
| 36 900 | 101 | 84 | 66 |
| Avg. 36 000 (machinery iron) | 103 | 77 | 61 |

MacPherran(36) reported results on bars up to 4 in. as shown below:

| Tensile Strength, 1.25-in. Diameter Bar, as Cast, lb. per sq. in. | Percentage of Tensile Strength of 1.25-in. Diameter Bar | | | | | |
|---|---|---|---|---|---|---|
| | $\frac{13}{16}$-in. Diameter Bar | $1\frac{3}{4}$-in. Diameter Bar | $2\frac{1}{4}$-in. Diameter Bar | $2\frac{3}{4}$-in. Diameter Bar | $3\frac{1}{4}$-in. Diameter Bar | 4-in. Diameter Bar |
| 21 100 | 134 | 80 | 64 | .. | .. | .. |
| 22 000 | ... | 77 | 64 | 66 | 68 | 64 |
| 31 000 | ... | 86 | 74 | 71 | 63 | 61 |
| 34 600 | 122 | 81 | 71 | .. | .. | .. |
| 54 000 | ... | 94 | 89 | 87 | 80 | 71 |

Some average results from a study of 37 foundry irons commercially produced are shown below:

| Class | Tensile Strength, per cent[a] | | | |
|---|---|---|---|---|
| | $\frac{3}{4}$-in. Diameter Bar | 1.1-in. Diameter Bar | 1.6-in. Diameter Bar | 2.0-in. Diameter Bar |
| 20 | 100 | 79 | 60 | 53 |
| 25 | 100 | 81 | 73 | 72 |
| 30 | 100 | 78 | 71 | 67 |
| 35 | 100 | 83 | 68 | 66 |
| 40 | 100 | 85 | 80 | 67 |
| 50 | 100 | 90 | 79 | 72 |

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[a] Private communication from Gray Iron Institute.

Data for some high-strength nickel alloy irons are given in Table XXVII.

All of these tests were made on a core machined from the "as cast" bar, and thus represent only a fraction of the original area and the weakest part of the bar. A truer picture of the actual relation

TABLE XXVII.—DATA FOR HIGH-STRENGTH NICKEL ALLOY IRONS (COYLE).[a]

| TOTAL CARBON, PER CENT | SILICON, PER CENT | NICKEL, PER CENT | TENSILE STRENGTH, 1¼-IN. DIAMETER ROUND BARS, LB. PER SQ. IN. | PERCENTAGE OF TENSILE STRENGTH OF 1¼-IN. ROUND BAR REMAINING IN METAL AS CAST IN 3-IN. ROUND BARS | |
|---|---|---|---|---|---|
| | | | | SURFACE | CENTER |
| 2.73 | 1.54 | 0.95 | 54 000 | 94 | 67 |
| 2.73 | 1.57 | 1.34 | 59 200 | 90 | 85 |
| 2.59 | 1.42 | 2.01 | 52 600 | 81 | 75 |
| 2.70 | 1.71 | 2.41 | 68 600 | 87 | 78 |
| 2.92 | 1.45 | 4.01 | 55 000 | 88 | 82 |

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[a] Private communication.

TABLE XXVIII.—EFFECT OF SECTION SIZE ON MODULUS OF RUPTURE (ROTHER AND MAZURIE).

| MODULUS OF RUPTURE ON 1-IN. DIAMETER BAR, AS CAST, LB. PER SQ. IN. | PERCENTAGE OF STRENGTH OF 1-IN. DIAMETER BAR | | | |
|---|---|---|---|---|
| | 1½-IN. DIAMETER BAR | 2-IN. DIAMETER BAR | 2½-IN. DIAMETER BAR | 3-IN. DIAMETER BAR |
| SOFT IRON | | | | |
| 49 000 | 96 | 88 | 80 | 76 |
| 53 000 | 93 | 84 | 83 | 78 |
| 56 800 | 95 | 88 | 86 | 84 |
| 52 900 | 95 | 87 | 83 | 79 |
| MACHINERY IRON | | | | |
| 69 400 | 95 | 96 | 92 | 87 |
| 71 000 | 94 | 94 | 84 | 81 |
| 70 200 | 95 | 95 | 88 | 84 |

TABLE XXIX.—EFFECT OF SECTION SIZE ON MODULUS OF RUPTURE (BORNSTEIN).

| MODULUS OF RUPTURE ON 1.2-IN. DIAMETER BAR, AS CAST, LB. PER SQ. IN. | PERCENTAGE OF STRENGTH OF 1.2-IN. DIAMETER BAR (TRANSVERSE) | | |
|---|---|---|---|
| | ¾-IN. DIAMETER BAR | 2-IN. DIAMETER BAR | 3-IN. DIAMETER BAR |
| SOFT IRON | | | |
| 41 800 | 133 | 94 | 78 |
| 43 000 | 121 | 91 | 76 |
| Avg. 42 400 | 127 | 93 | 77 |
| MACHINERY IRON | | | |
| 58 400 | 115 | 95 | 80 |
| 60 900 | 113 | 100 | 94 |
| 65 000 | 117 | 97 | 82 |
| Avg. 61 400 | 115 | 97 | 85 |

of section size to strength is shown by the transverse test, which is primarily a test of the outer part of the bar, which is the major portion of the material. For example: Table XXVIII shows the percentages obtained by Rother and Mazurie(34) on the "as cast" bars tested on 12-in. centers.

The real value of the heavier sections is clearly shown, the soft iron showing only 21 per cent drop from 1 in. to 3 in. instead of the 45 per cent drop given by the tension test in the data given by Rother and Mazurie.[1] The machinery iron also shows much better values throughout.

The transverse data of Bornstein(35), shown in Table XXIX, substantiate those of Rother and Mazurie. The difference between transverse and tension values is notable.

The point illustrated in Fig. 22, that the ratio of modulus of rupture to tensile strength varies inversely as the tensile strength, is a partial explanation of this difference, but the difference in structure of the tensile specimen (the inside core) and the much greater area of stronger metal (the outside) which functions in transverse loading is responsible for the greater part of it.

It is apparent from all of these results that the effect of section size is not so pronounced on the stronger as on the weaker irons. Also most results show a definite flattening of the loss of strength curve as the large sizes of bars are reached.

None of the above results show it, but there is a peak which is peculiar to each iron—in other words rapid cooling increases strength up to a certain point, after which the decrease is sometimes quite rapid. This is usually where massive cementite is formed (the iron begins to mottle). Pearce(37) shows (Fig. 33) this phenomenon on three cast irons differing in analysis. Irons beyond the peak are not true gray irons.

Since the other properties treated in this section have, in a statistical manner, been correlated with the tensile strength, it is not necessary to show their relation to section size, as, in general, their relation to tensile strength is the same without regard to the method of producing the tensile strength.

*Damping Capacity:*

Damping capacity, only recently brought prominently to the attention of designing engineers, is defined as the amount of work dissipated into heat by a unit volume of the material during a completely reversed cycle of unit stress or, in other words, the ability to

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[1] For a summary of this data, see table on p. 190.

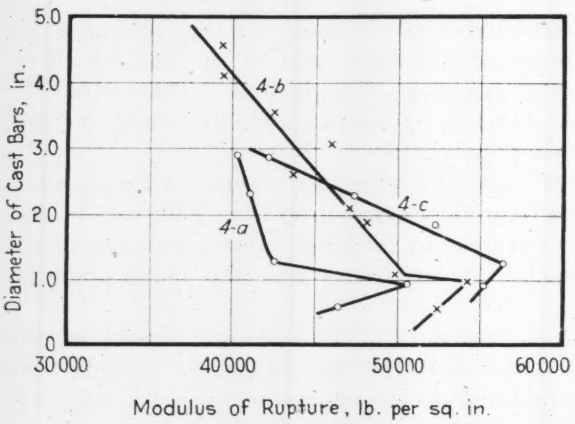

FIG. 33.—Rupture Moduli of Large Bars Showing Peak. (Pearce.)

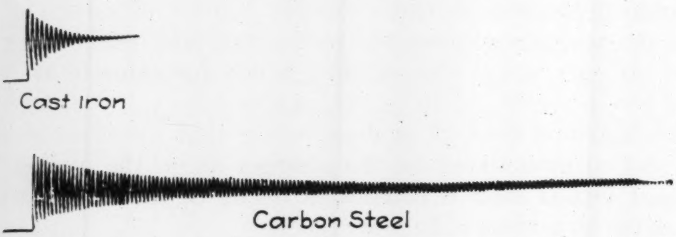

FIG. 34.—Curves Taken with Foeppl-Pertz Damping Tester Indicate the Difference Between Internal Damping Capacity of Cast Iron and Carbon Steel. (Pohl.)

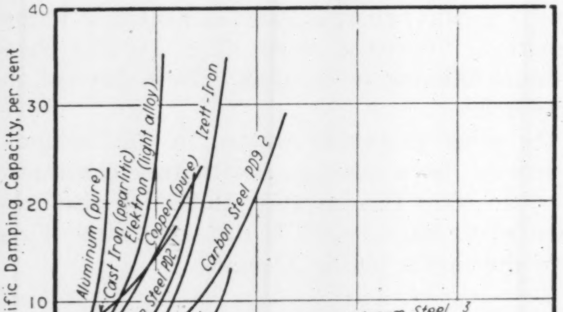

FIG. 35.—Damping Characteristics for Materials of Comparatively High Damping Capacity. (G. S. von Heydekampf.)

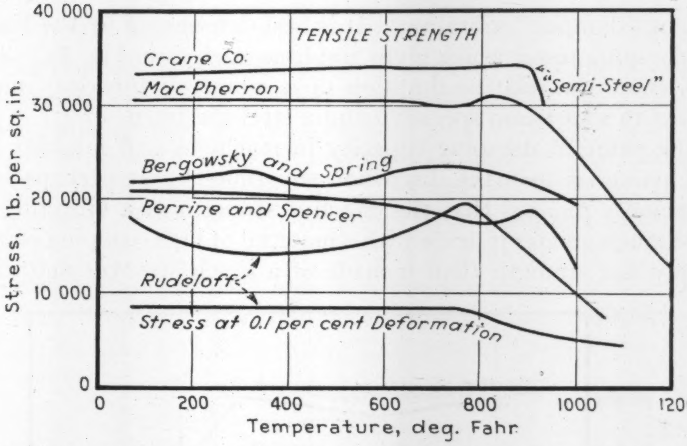

FIG. 36.—High-Temperature Tensile Properties of Cast Iron and Semi-Steel as Reported by Various Investigators. (H. J. French and W. A. Tucker.)

| Investigator | Chemical Composition, per cent | | | | | | | Remarks |
|---|---|---|---|---|---|---|---|---|
| | Combined Carbon | Graphitic Carbon | Total Carbon | Manganese | Silicon | Phosphorus | Sulfur | |
| MacPherran.... | .... | .... | .... | 0.64 | 1.84 | 0.52 | 0.11 | Annealed at 1100 F. (595 C.) before test. |
| Bregowsky and Spring....... | 0.17 | 3.31 | .... | 0.60 | 2.57 | 0.73 | 0.10 | |
| Perrine and Spencer...... | .... | .... | 2.69 | .... | .... | .... | .... | Curve based on very few tests. |
| Rudeloff[a]....... | .... | .... | 3.56 | 0.93 | 2.64 | 0.52 | 0.05 | Curves are averages from both wet and dry sand castings. |
| Crane Co....... | "Seim-steel" or "ferro-steel" | | | | .... | .... | .... | |

[a] 1000 lb. per sq. in. = 0.7031 kg. per sq. mm.

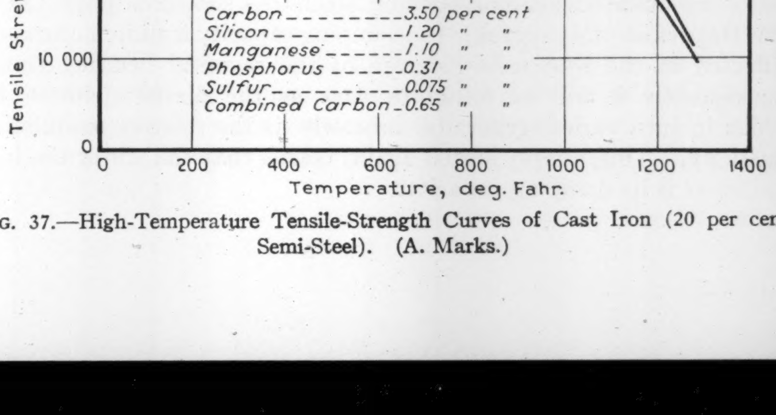

FIG. 37.—High-Temperature Tensile-Strength Curves of Cast Iron (20 per cent Semi-Steel). (A. Marks.)

absorb or "dampen" vibration. It is best determined by the Foeppl-Pertz damping tester which gives graphs(38) such as in Fig. 34. These graphs show the relative duration of a vibratory torsional impulse imparted to a cast-iron specimen and a steel specimen.

The value of damping capacity in machines and machine tools is so obvious as to make discussion superfluous. It perhaps is not so commonly realized that the effective strength of a vibrating part may be much greater if made with a material of high damping capacity and only fair strength than if made of a much stronger material of low damping capacity—the latter allowing vibrations to build up to a serious intensity while the former will "damp" them out.

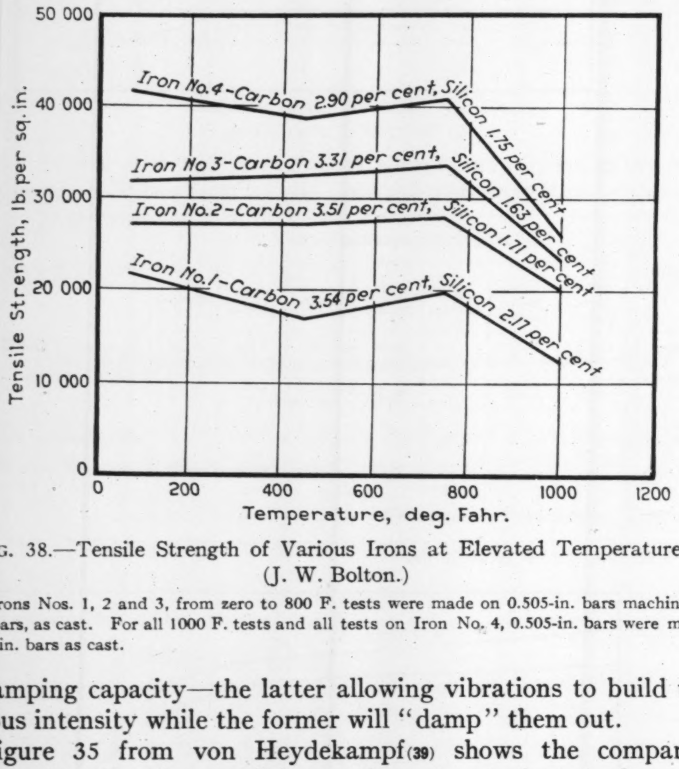

FIG. 38.—Tensile Strength of Various Irons at Elevated Temperatures. (J. W. Bolton.)

In Irons Nos. 1, 2 and 3, from zero to 800 F. tests were made on 0.505-in. bars machined from 1.25-in. bars, as cast. For all 1000 F. tests and all tests on Iron No. 4, 0.505-in. bars were machined from 1.0-in. bars as cast.

Figure 35 from von Heydekampf(39) shows the comparative damping capacity of a pearlitic cast iron, two carbon steels, a brass and a chromium-nickel-molybdenum steel. As shown on page 159 of von Heydekampf's paper(39), the damping capacity is quite accurately reflected in the hysteresis diagram of the reversed bending test—consequently it will be found to vary as the plastic deformation which in turn varies (generally) inversely as the relative modulus of elasticity. This, simply stated again, means that the stiffer the iron the lower is its damping capacity.

*Acoustics:*

The acoustic properties are also a function of the elastic modulus. The acoustic resistivity is given (International Critical Tables) as 255 for ordinary cast iron as compared to 390 for steel and 232 for brass. The velocity of sound waves in cast iron are given in feet per second by the same authority as 12,000 against 16,500 for steel and 9000 for brass. This velocity decreases with rising temperature.

The formula for acoustic resistivity is given as $u = \sqrt{Ep}$, where $E$ is modulus of elasticity and $p$ is the specific gravity. As both quantities decrease with rising temperature, the resistivity also decreases as the metal is warmed. The acoustic properties are closely related to the damping capacity since the duration of sound from a vibrating body is that portion of the damping graph which registers on the ear.

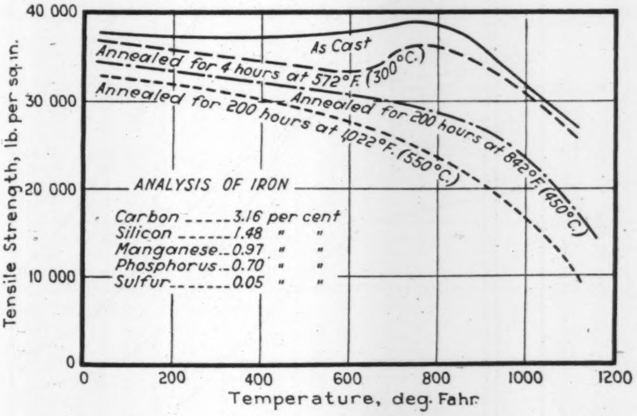

FIG. 39.—Tensile Strengths of Cast Iron at Elevated Temperatures. (J. W. Donaldson.)

*Effects of Elevated Temperatures* (40):

*Short-Time Tension Tests.*—Gray irons, irrespective of classes or grades, when judged by short-time tests, appear to have a very slight change from their original tensile strengths between room temperature and 800 F. In certain cases a slight increase may appear between 600 and 800 F. Above 900 F. there is sharp lowering in strengths in all grades, which lowering continues with increase in temperature. Gray iron exhibits quite constant tensile strengths up to or slightly above 800 F. on short-time tests. This is indicated by Figs. 36, 37, 38, 39 and 40.

*Fatigue or Endurance Limit.*—Endurance limit is lowered little, if any, up to 800 F., and above this temperature the ratio of endurance limit to tensile strength increases. Above 1200 F. the endurance limit apparently is fully as high as is the tensile strength under steady prolonged load, according to Moore and Lyon(41).

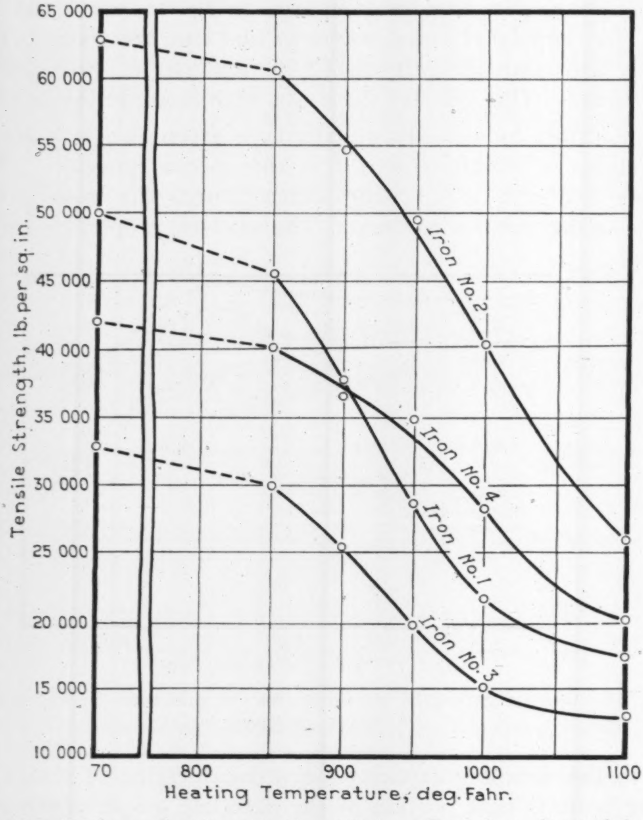

FIG. 40.—Tensile Strength After Heating 500 hr. Tested at elevated (annealing) temperatures.

| | Tensile Strength, lb. per sq. in. | Chemical Analysis, per cent | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Total Carbon | Silicon | Phosphorus | Manganese | Sulfur | Nickel | Chromium |
| Iron No. 1 .......... | 49 900 | 2.96 | 2.53 | 0.06 | 0.78 | 0.11 | 0.96 | 0.02 |
| Iron No. 2 .......... | 62 700 | 2.67 | 2.39 | 0.07 | 0.67 | 0.09 | 0.76 | 0.45 |
| Iron No. 3 .......... | 32 800 | 3.63 | 1.15 | 0.17 | 0.54 | 0.09 | 0.03 | 0.03 |
| Iron No. 4 .......... | 41 900 | 3.46 | 1.36 | 0.39 | 0.68 | 0.08 | 0.01 | 0.54 |

*Brinell Hardness.*—Brinell hardness is unchanged up to 800 F. according to all tests found in the literature.

*Relative Modulus of Elasticity*[1].—Stress-strain diagrams indicate lowered relative modulus of elasticity at elevated temperatures. Figure 41 shows stress-strain curves for a high-test iron as determined at room temperature (70 F.), 450, 750 and 1000 F. A given stress produces a greater elongation, hence a lowered relative modulus of elasticity, as the temperature is increased. The strength and relative modulus of elasticity are lowered as section size increases.

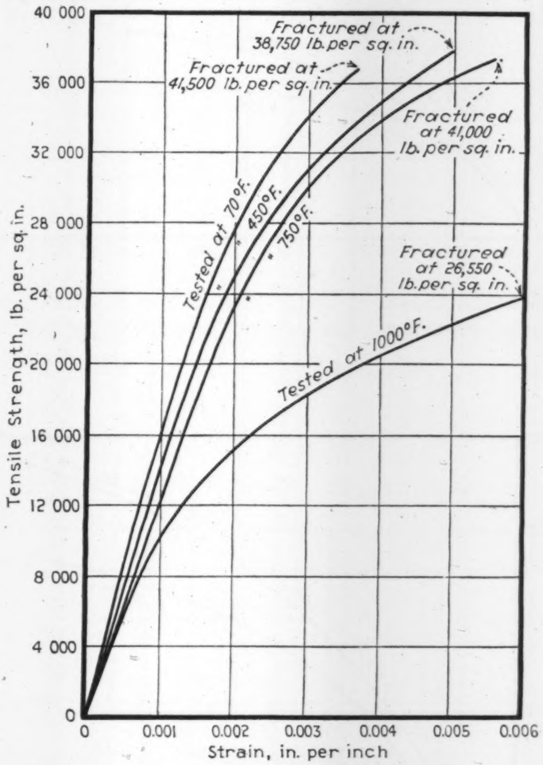

FIG. 41.—Stress-Strain Curves for High-Test Iron of the Following Composition: 2.90 per cent Carbon, 1.75 per cent Silicon, 1.15 per cent Nickel.

Test specimen 0.505 in. in diameter taken from bar 1 in. in diameter, as cast.

In Figs. 42 and 43 are shown stress-strain diagrams from tests conducted at 1000 F., on different types of iron, and on bars of various diameters as cast. As noted on the charts these stress-strain diagrams

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[1] Since the stress-strain diagram of gray iron exhibits curvature almost from the beginning, there exists no constant proportionality between stress and strain, hence no fixed modulus of elasticity. The term "relative modulus of elasticity" is used to denote qualitatively the relative steepness of the stress-strain curves.

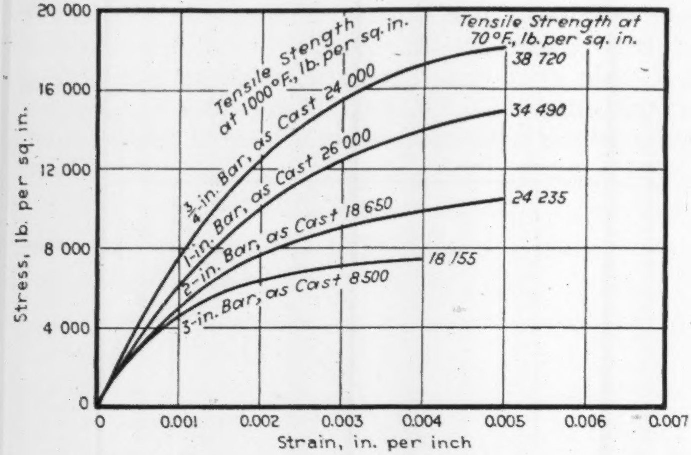

FIG. 42.—Stress-Strain Curves of Tests Made at 1000 F. on an Iron Having the Following Composition: 3.25 per cent Carbon, 1.70 per cent Silicon.

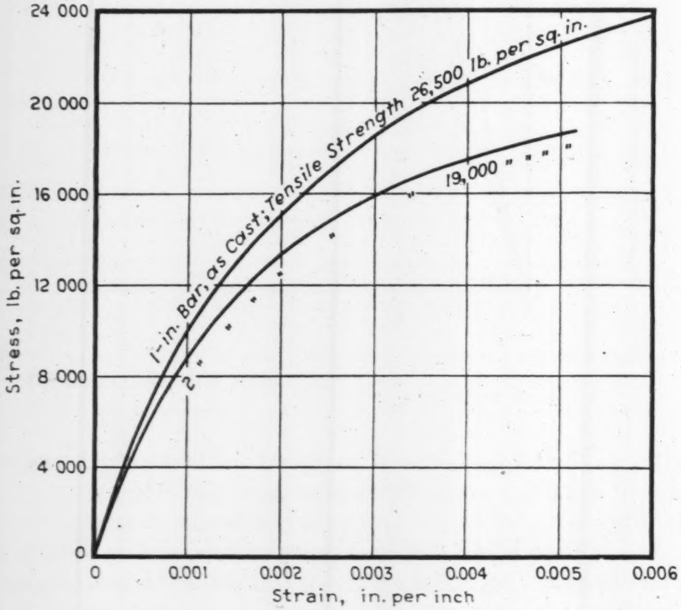

FIG. 43.—Stress-Strain Curves for "High-Test" Iron Having the Following Composition: 2.90 per cent Carbon, 1.75 per cent Silicon, 1.15 per cent Nickel.

Machined to 0.505 in. in diameter test specimens and tested at 1000 F.

are incomplete, the extensometer being removed before breaking of the specimen.

Two characteristics are notable: (1) The stress-strain diagrams for different irons tested at elevated temperatures place these irons in the same relative order as do room temperature tests, that is, the irons, which are stronger and possess greater relative elastic moduli, also are stronger and have higher elastic moduli at 1000 F. (Figs. 41 and 43). (2) A given class or grade of iron shows lowered strength and lowered relative elastic modulus at a given temperature as the test bar size is increased—which is comparatively the same phenomenon exhibited at room temperature.

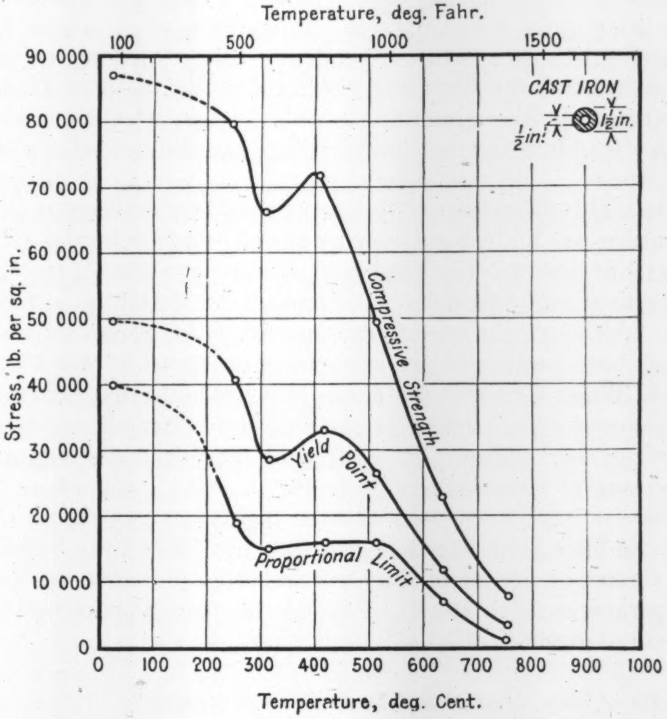

FIG. 44.—Compressive Strength of Cast Iron Tested at Constant Temperatures.

Those interested in design must not forget that the above are short-time tests, not long-time tests.

*Effect of Compression.*—The relation of compressive strength to temperature is shown in Fig. 44, according to Ingberg and Sale(42). The figure is self-explanatory. The iron tested was of rather poor grade, with 3.52 per cent carbon, 1.58 per cent silicon, and 0.72 per cent phosphorus.

*Wear Resistance.*—That the wear-resisting properties of gray irons at various temperatures are usually good has been well known to engineers for many years. The satisfactory employment of gray iron in automotive, steam and Diesel engines is known to all. A reason for the good wear resistance of gray irons lies in their relative freedom from tendency to gall or seize. This was demonstrated by Mochel(43), who tested various combinations of metals at 750 F., and determined their relative tendency toward galling or seizing. The cast irons all operated well, both in contact with cast iron and also with metals like monel and stainless irons, which latter metals seize very readily when run on themselves but did not seize when run on cast iron.

*Limits of Application.*—The uses of gray-iron castings at elevated temperatures are limited not only by the physical properties as shown by tests but also by the tendency of gray-iron alloys to "grow." The phenomenon "growth" is discussed in detail in a following section. Although the use of gray-iron alloys for pressure-containing parts has been limited to a maximum temperature of 450 F. by the A.S.M.E. Boiler Construction Code, it is probable that this rating is unduly severe when applied to some of the better classes or grades or cast iron used today. In other fields gray-iron castings are frequently used at temperatures up to 700 or 800 F., and where service is not severe have been used at even higher temperatures. Where service conditions indicate that growth is not likely to be serious, the better classes or grades of gray-iron castings probably can be used at temperatures up to 800 F. Beyond this point it appears that the likelihood of graphitization will limit their use.

### *Properties of Gray Iron at Subnormal Temperatures:*

Exposure of gray irons to subnormal temperatures apparently does not result in serious impairment in physical properties. Tensile strength and Brinell hardness are maintained, or slightly increased, according to tests made at various temperatures down to −317 F. At temperatures of −112 F. and below there is a slight decrease in impact value. There is no pronounced increase in brittleness or change in appearance of fracture at −317 F. (A soft steel, originally with 23.5 per cent elongation, showed only 4 per cent, with a brittle-appearing fracture, when broken at −317 F.)

Design for low-temperature service conditions should allow for thermal contraction and the consequent possibility of internal stresses.

*Growth of Iron:*

The term "growth" as applied to gray cast irons refers to the permanent increase in volume which sometimes occurs when the metal is subjected to repeated cycles of heating and cooling. Stresses from external sources are not necessary to cause growth. Under ordinary circumstances, serious and continued growth rarely occurs at temperatures well under 1000 F. However, in the presence of certain corroding agents, such as superheated steam for example, growth of certain grades of iron may occur at much lower temperatures.

Growth, as the term implies, results in permanent increase in volume, in extreme cases up to 50 per cent or more. It also causes very serious loss in strength and great brittleness. Specific gravity is lowered. In severe cases of growth, the metal retains negligible tenacity and is worthless as an engineering material.

The cause of permanent or irreversible growth in gray iron may be summarized under the following headings:

(*a*) Expansion due to graphitization.
(*b*) Corrosion.
(*c*) Thermal gradients.
(*d*) Allotropic changes (involving expansion and contraction).
(*e*) Pressure from occluded gases (this point is disputed by many).

These factors generally act in an interdependent manner and most of them are progressive and mutually cumulative.

For convenience in exposition, we shall consider three types of cycles of heating and cooling:

(1) Below any ordinary graphitization range, say, up to 700 or 800 F.

(2) Within the range of graphitization but below the allotropic modification, say, 900 to 1300 F. for ordinary gray irons.

(3) Through and above the allotropic modification range, say, 1325 or 1340 F. and up.

Within the first range, causes or factors (*a*) and (*d*) are not involved. Referring to Piwowarsky's work (which may be considered typical) samples heated in rape-seed oil (and thus protected from corrosion) showed no growth up to 570 F. However, when heated in steam for 75 hr., growth took place in the coarse-grained irons, and at 660 F. there was corrosion even to the interior of the coarse-grained irons. The fine-grained irons did much better as the internal corrosion in steam was very slight, even at 840 F. This is the general experience of users of gray iron for valves, piping, fittings and other pressure-containing parts for superheated and higher-pressure steam. With dense sound castings and good design, growth may not be a serious factor up to 600 or 700 F. If porosity or open grain allow penetration of oxidizing gases, then trouble is likely. However, it is not alone the possibility of growth that discourages the use of gray iron for superheated, high-pressure steam.

The above shows that corrosion is an important factor in the range from room temperature to, say, 800 F. The products of corrosion occupy greater volume than the original metal and destroy its tenacity. A dense structure prevents penetration, hence, hinders growth.

Thermal gradients promote cracking. For example, the outer layers of a piece being heated come up to temperature faster than the interior and consequently are expanded more until uniformity of temperature is reached throughout the casting. This results in stresses which are likely to cause cracks. Reversing, on cooling, the rapidly contracting exterior tries to compress the still expanded interior, resulting in more stresses and possibly more cracks. Remembering that gray iron is a relatively non-ductile material, even though visible cracks may not be evident, certain rapid rates of cooling and heating undoubtedly result in formation of many incipient cracks. These cannot knit together and actual growth may be a result. Furthermore, the cracks also allow a better chance for infiltration of corroding media. It might be mentioned that the expansion of occluded gases, if present, may possibly result in localized stresses and incipient cracks.

In the second range, we have all the factors operative in the first range—intensified in many respects by the increase in temperature. Perhaps the only ameliorating factor is the increased plasticity or ductility of the metal in the higher temperature range. However, we have the new factor, graphitization. The thermodynamically stable condition of the iron-carbon alloys in the gray-iron range is ferrite-graphite. The pearlite is unstable; it is always ready to break down to graphite and ferrite—which occupy more volume than the original pearlite. Below 800 F., the rate of change of pearlite to graphite and ferrite is so extremely slow that it usually is not of practical importance. There are some evidences of graphitization at 900 F. and from this point on there is a rapid increase in rate concurrent with increase in temperature. Above the transformation range, 1340 F., there is some resolution of graphite, which increases with increase in temperature. Thus in the range 900 to 1300 F., the growth rate increases with temperature because of the graphitization, as well as because of the intensified action of corrosive media and the likelihood of greater thermal gradients.

At or about 1340 F. there occurs an allotropic transformation which causes a marked change in volume. At this temperature the pearlite-ferrite mixture (alpha ferrite and cementite) changes to austenite (gamma ferrite and dissolved carbide). This volume change in castings with thermal gradients may quite readily produce incipient fractures. Also, most of the other factors promoting growth have their actions intensified by the higher temperature. The corrosion rate is increased, graphitization is much more rapid and pressure from possible occluded gases is increased. The plasticity is greater at the higher temperatures which may partially counteract the tendency toward cracking.

*Control of Growth.*—Let us consider first the regular (not high-alloy) gray irons. Obviously those of finest "grain," densest, with lowest coefficient of expansion and "chemically" most stable will best resist growth.

White iron containing no graphite is graphitized with much greater difficulty than gray iron which is partially graphitized to start with. White iron, therefore, is more resistant to growth.

Silicon, promoting graphitization, increases growth at temperatures above about 900 F. Other elements such as aluminum and nickel promote growth. However, the substitution of nickel for silicon may diminish growth, in this range nickel being a less powerful graphitizer and its graphitization reaction stopping at or about the eutectoid ratio. At high temperatures, silicon in the silico-ferrite apparently is readily oxidized to silica ($SiO_2$), which occupies a large volume. This in itself may be a factor in growth.

Chromium, because of its ability to effect the stabilization of carbide, is usually one of the most effective elements for retarding growth. (Tungsten and molybdenum quite likely have a similar effect.) Manganese and sulfur in themselves are carbide stabilizers, but one neutralizes the other. High sulfur and low manganese, or the converse, appear desirable. High phosphorus, forming steadite envelopes, retards penetration of corrosive agents and hence is beneficial in retarding growth.

Low-total-carbon irons, if fine grained and with stabilized carbide, can be expected to be more growth-resistant than higher carbon irons with heavy graphite flakes.

Dense grain, effected by superheating, correct composition, proper cooling rate, etc., is desirable.

Good molding practice, hot iron and good feeding to produce really sound castings are necessary.

Care in design will do much to avoid trouble from thermal gradients. Heavy sections are likely to be coarse-grained, and certainly are more likely to have thermal gradients. Non-uniform sections cause thermal gradients, internal stresses and trouble.

The effect of occluded gases does not seem to be proved to the satisfaction of all.

It is possible to avoid the allotropic transformations by the use of high-alloy irons.

Special irons for corrosion-resistant uses are covered in the section on Corrosion Resistance of Cast Iron.[1]

In concluding, it may be pointed out that the troubles encountered from growth often have been due to employment of grades of iron unsuited for the service conditions. The A.S.M.E. Boiler Construction Code does not permit the use of cast iron for pressure-containing parts at temperatures over 450 F. Yet it would not be difficult to find many places where suitable grades of iron have given many years of satisfactory service at somewhat higher temperatures. While the use of gray iron beyond its rightful sphere is not to be encouraged, it should be emphasized that the use of better grade of irons, more widely available today than heretofore, may justify a somewhat greater latitude in application.

*Machineability of Cast Iron:*

Machineability of metals has been variously determined with reference to (*a*) tool life, (*b*) type of finish obtained, and (*c*) power required. Since differences in machineability with reference to tool life is of the greatest practical importance, most of the following statements are confined to this phase of the subject with brief reference to machineability in terms of surface finish. References to articles by various investigators on this general subject are given in the bibliography, references (44) to (50), inclusive.

The composition, structure and physical properties of cast irons extend over a wide range, and dependent upon these factors, the machineability of cast irons varies from being one of the most readily machineable to the most difficult among ferrous materials. At one extreme of this range is iron made in permanent molds and then completely annealed which is the most easily machineable, and at the other extreme, there is white or chilled cast iron. Annealed permanent-mold cast iron possesses excellent machineability because its structure consists of iron containing no combined carbon and broken up by a high percentage of finely divided free carbon flakes together with freedom from burned-in sand at the surface. Chilled or white iron is extremely difficult to machine because its structure is composed chiefly of hard iron carbide in both pearlitic and massive form unbroken by any flakes of free carbon. The varying degrees of machineability possessed by types of cast iron in between these two extremes depends upon the proportions present and the arrangement of hard and soft microstructural components of cast iron. The various microstructural constituents of cast irons may be listed in the order of their hardness somewhat as follows, starting with the softest:

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[1] See p. 215

(*a*) Free carbon (graphite or temper carbon in flake form).

(*b*) Silico-ferrite (iron with silicon in solution).

(*c*) Silico-ferrite alloyed with other metals which dissolve in iron.

(*d*) Pearlite—alternate laminations of iron and iron carbide.

(*e*) Alloy pearlite.

(*f*) Sorbite.

(*g*) Steadite (iron phosphide)—non-metallics such as sand burned into the surface or included within the iron, manganese sulfide, silicates, etc.

(*h*) Massive cementite (iron carbide, manganese carbide, chromium carbide).

It will be appreciated at once that the possible number of different proportions and arrangements of the above constituents would be accountable for any degree of machineability between the two extremes cited above. For a more detailed discussion of these variations, it is desirable to classify the different cast irons according to their microstructure, as:

(*a*) Ferritic—composed of free-carbon and silico-ferrite.

(*b*) Pearlito-Ferritic (free carbon, silico-ferrite and pearlite).

(*c*) Pearlitic (free carbon and pearlite).

(*d*) Mottled iron (pearlite and enough massive cementite to produce a mottled fracture).

(*e*) White iron (pearlite and massive cementite).

*Ferritic Cast Iron.*—Ferritic cast irons are usually low strength soft cast irons and are quite easy to machine. Slight variations in ease of machining in this group are due to:

(*a*) Increasing total carbon—improves machineability.

(*b*) Increasing silicon content—decreases machineability.

(*c*) Fineness of free carbon particles—improves machineability.

(*d*) Residual cementite undecomposed by annealing or steadite in small quantities would have negligible effect but if these were in sufficient quantities throughout or in segregated masses as might occur in the neighborhood of shrinks, the machineability would be adversely affected.

*Pearlito-Ferritic Cast Irons.*—Cast irons in this group may vary from weak soft irons of about 130 Brinell hardness, containing only a small amount of pearlite, up to fairly hard irons of medium strength; for example, up to 220 Brinell hardness and 38,000 lb. per sq. in. tensile strength in which a pearlitic microstructure predominates with only small amounts of ferrite.

The machineability in this group will be approximately inversely proportional to tensile strength, both being influenced by the percentage of pearlite and total carbon present. Those with high total carbon and low pearlite content will machine similarly to those in the ferritic group. On irons at the other extreme of this group with low total carbon and small amount of free ferrite, especially if made by one of the processes producing an arrangement of graphite flakes resulting in high strength, the machineability will be considerably less.

In this group of irons the presence of massive carbides or steadite will result in rapid failure of cutting tools because the presence of the pearlite will anchor the hard carbides or steadite in place, making it necessary for the tool to cut through these hard particles rather than tearing them out as in soft ferritic irons.

In irons of equal total carbon content subjected to a certain cooling rate, increase in silicon will tend to decrease the pearlite content (combined carbon) and increase the graphitic carbon content, thus improving the machineability. On the other hand, by varying the cooling rate to produce a constant amount of pearlite and graphitic carbon in irons of different silicon content, the higher silicon irons will be more difficult to machine on account of the hardening effect of the silicon upon the iron in which it dissolves.

*Pearlitic Irons.*—Irons in this group will have machineability similar to the irons in the pearlito-ferritic group, having a small amount of free ferrite. Massive carbides and steadite in irons of this group will be ruinous to cutting tools. Assuming freedom from massive carbides and steadite, machineability will be more difficult with increasing hardness and tensile strength. Alloys may be used for producing greater resistance to wear or strength in cast irons. These will affect the machineability in so far as they form or prevent the formation of carbides, affect the strength or hardness of the iron.

*Mottled Iron.*—Due to the considerable amounts of massive iron carbide in mottled iron, it is machineable only at greatly reduced speeds. Few commercial uses are made of this type of iron.

*White Iron.*—White cast irons are naturally the most difficult to machine. Where machining of white iron must be done, it is carried on at very slow speed as in the case of chilled rolls. In most cases where white iron is machined after casting, it is done by grinding. The lower the total carbon content of white cast irons, the less difficult it is to machine them.

*General.*—Under certain conditions it is possible to obtain in one casting, structures falling in two or more of the groups discussed above. As an illustration, an automobile cylinder block casting having light and medium sections in which the highest possible hardness is desired in the cylinder bore, compatible with ability to machine the faster cooling lighter sections. To obtain this in a plain cast iron with about 3.25 per cent total carbon, it is necessary to use about 2.00 to 2.40 per cent silicon. The hardness of the cylinder bore will be between 160 and 180 Brinell. If it is attempted to increase this hardness to 200 Brinell by decreasing the silicon content of the iron to, say, 1.80 per cent, the light sections and corners will have white iron spots composed principally of massive iron carbide which is practically unmachineable. One way in which the hardness of cylinder bores has been increased without incurring hard spots in the flanges and corners has been to raise the silicon to about 2.75 per cent and adding 0.50 per cent chromium. A more common method, however, is to lower the silicon content and add nickel or nickel and chromium. The nickel has the power to prevent the formation of massive carbides in the rapidly cooled sections and, at the same time, it increases the hardness of the pearlitic matrix. By adding 1.00 to 1.50 per cent nickel and about 0.50 per cent chromium, a cylinder bore hardness of 225 to 250 Brinell can be obtained in a cylinder block casting which is at the same time readily machineable even in the light sections.

The action of nickel is to prevent the formation of white iron in light sections, thus improving machineability. When it is used with lower silicon content or with lower silicon and with chromium to raise the hardness, it permits the machining of irons of considerably higher hardness than could be machined without the use of nickel, although the degree of machineability of these alloy cast irons will be approximately inversely proportional to the hardness.

The ability to machine irons of 225 to 250 Brinell is due to the fact that no massive carbides exist in the matrix. As soon as a plain cast iron exceeds a hardness of 200 Brinell, it usually contains, especially at the edges and corners, numerous carbides and other hard spots which cause early failure of cutting tools. By "other hard spots" is meant a structure frequently observed in cast iron occurring in a broad network form having the appearance of sorbite containing a high percentage of combined carbon in solution. Massive carbide spots are frequently located within the boundaries of this network.

Chromium alone forms chromium carbides so that if as much as 0.25 per cent is addied to a plain cast iron which already is at the limit of machineability, the iron will then become practically unmachineable. It is customary therefore to use chromium only in combination with an element which prevents carbide formation such as nickel or increased silicon.

Molybdenum frequently is used as an alloy for cast iron to obtain certain strength and wear resistance characteristics. This element does not prevent carbide formation but where it is used to increase hardness and strength without formation of carbides, the machineability will be inversely proportional to the hardness or strength produced.

*Surface Conditions.*—The machining of all cast irons where skin cuts must be made is rendered difficult by the presence of any burned-in molding sand. Where burned-in sand is present, any attempt to make light surface cuts will cause rapid failure of the tool. A sufficient depth of cut must be taken to insure the point of the tool being well below the surface of the casting. The more efficient is the mold facing used in preventing the fusing of molding sand to the surface of the casting, the better will be the tool life. One of the reasons for the superior machineability of permanent mold castings is the entire absence of any burned-in sand on the surface of the casting.

In addition to burned-in sand at the surface, some castings exhibit an extremely thin surface layer of white iron. This thin layer might escape detection unless the fracture of the casting is examined very closely. Such a layer will obviously cause rapid tool wear. This thin white layer may be due to the molding sand being too wet or to improper analysis. Alloying methods discussed above have been found effective in removing it.

*Machining Finish.*—The character of the machined surface, its accuracy and appearance are influenced by the depth of cut, feed, shape of cutting point of the tool, the speed at which the metal is cut and upon the rigidity of the cutting tool, the machine tool, the mounting of the work in the machine and the flexibility of the casting itself. Accuracy of finish will also depend upon the quality of the tool used, being least with carbon tool steel and greatest with the carbide type of tools.

Any possibility of movement of the casting or the tool, other than that necessary for producing the cut, will result in inaccurate machining and chattering. It is necessary, therefore, to have the machine tool in good condition with no looseness in bearings or in tool mountings. The tool must be clamped in the tool mounting with as small an overhang as possible between the tool support and the cutting edge to reduce deflection of the tool to a minimum. The tool bit itself should be of sufficient size to avoid springiness. The casting being machined should be clamped to the machine tool bed or face plate in such a manner that there is the least possible chance for deflection of the casting between the place of clamping and the surface being machined.

The shape and section size of some castings make it almost impossible to prevent a certain amount of spring or give in the casting, even when light cuts are taken, especially in the case of turning operations. An example of this is in turning large-diameter brake drums. In this class of work, chattering is frequently encountered due to the vibrations set up in machining. An expedient which has been found successful in preventing this chatter is to stretch a coil spring around the outside circumference of the drum. This apparently acts to counteract the vibrations caused by cutting, thus eliminating the chatter. By proper design of tool to produce greatest freedom of cutting, it is often possible to eliminate the necessity for this spring.

The character of the surface finish obtained in machining cast irons under suitable cutting conditions, as regard rigidity of tool, work and machine, is then principally dependent upon the size of the graphite flakes and the total amount of free carbon. A high total carbon, high-silicon iron, cast in heavy sections with the attendant slow cooling in the mold so that it contains large graphite flakes in such quantities that they almost intersect one another, will show a pitted surface when machined due to tearing out of particles of metal between the graphite flakes. High-carbon, high-silicon iron cast in thin sections in sand or in permanent molds will possess finely divided graphite and a close grain which will give a very smooth, dense-appearing, machined surface.

Rough cuts taken on coarse-grained cast iron will exaggerate the pitted appearance by tearing out small particles of iron. Even when the finest cut is taken on this type of iron, it will have an open appearance due to the large size and amount of graphite flakes. Certain types of cast iron in thick sections will show a close grain at the surface where the graphite is fine and the grain size small, with increasing grain size the greater the depth below the surface. Light cuts on this type of material will produce a good surface finish but deeper cuts will reveal the coarser structure. Difference in cooling rate from one part of a casting to another will cause variation in grain size and consequent change in surface appearance after machining.

*Wear Resistance of Cast Iron:*

Wear may be termed the mechanical or physical surface disintegration due to moving contact with another metal or other material. This process results in the removal of surface layers of the metal, causing a dimensional change which affects the functioning of the part involved and if continued will eventually result in complete failure.

The wide use of cast iron for parts of machinery which must resist wear is evidence that this metal is highly esteemed for such purposes. On account of the wide variation in properties of cast iron, depending upon chemical composition and conditions attending its manufacture and casting, the possibility of not obtaining the best results from cast iron exists especially for those who are unacquainted with certain more or less well-established principles governing its application to parts intended to resist wear.

From the standpoint of utilization to resist wear, cast irons may be divided into two general classes: (1) White or chilled irons and (2) gray irons.

*White or Chilled Irons.*—Under conditions of high unit load application and relative motion between two moving members, white or chilled irons have demonstrated marked superiority over gray cast iron. In such cases the high hardness of the white iron is necessary to prevent surface indentation. Typical examples are:

(1) High unit pressures imposed on tools during shaping and deforming soft ductile metals.

*Examples:* Wire drawing dies, chilled iron rolls, and dies for deep drawing sheet metals.

(2) High unit pressures attending line or small area of contact between two members.

*Examples:* Car wheels, cams, and cam followers.

(3) Scrubbing or impingement by abrasive materials.

*Examples:* Plow points, sand blast nozzles, sand pump castings, and centerless grinder work rests.

The composition of these castings varies with size of the casting and the depth of chill required. Alloys for obtaining special results may be used. The addition of chromium assists in obtaining the extreme hardness desired at the surface.

*Gray Cast Irons.*—Where contact pressures are not so high and larger areas in contact are involved, the danger of surface indentation is not present but a tendency to gall exists. In such cases the gray cast irons are valuable due to the graphite flakes distributed throughout the metal which serve to prevent galling. The least tendency toward galling is possessed by cast irons with the highest content of graphitic carbon, but such irons possess a low tensile strength and rapid wear occurs due to surface attrition. The properties in gray cast iron which are associated with low rate of wear are eutectoid combined carbon content and high hardness together with high strength. Other things being equal, an iron possessing a matrix of pearlite will resist wear better than one containing some free ferrite. Of two irons containing a matrix of pearlite, the one with the lower percentage of total carbon will have the better resistance to wear. This is because the distance between graphite flakes is greater in the iron containing the lower total carbon with the result that there is more resistance to picking out of minute particles from the surface.

Statements have appeared in the literature to the effect that there is no relation between Brinell hardness and resistance to wear. In special cases, this undoubtedly is true. For example, a very high-carbon, high-silicon iron cast in a metal mold and annealed will show a higher hardness than a low-carbon, medium-silicon iron cast in a sand mold and cooled slowly, but the permanent mold cast iron will wear much more rapidly than the sand-cast iron. Practice has shown, however, that other things being equal, higher hardness obtained by (1) lowering the total carbon, (2) increasing the pearlite content, (3) addition of alloys such as chromium, nickel, molybdenum and others, results in increased resistance to wear.

This has been demonstrated many times in brake drum tests comparing the wear resistance of drums made of ordinary cylinder iron having a hardness of about 180 Brinell, with drums made of low-carbon high-strength alloy cast iron having a hardness of about 260 Brinell. The 180 Brinell iron drums wear much more rapidly than the 260 Brinell iron. Many other service tests have given similar results.

Passenger car cylinder blocks are made from a gray iron which will result in a cylinder bore hardness of about 180 Brinell. With proper lubrication, the wear in this type of service is very slight, being from 0.001 to 0.002 in. on the bore in 20,000 miles of operation; but in bus and truck engines where the service may be more severe and number of miles operated greater, a hardness of 180 Brinell is not adequate to prevent excessive wear before the expected life of the engine is attained so that cylinder-block iron is used which will give from 210 to 260 Brinell and in some cases where even this hardness is not sufficient to hold the wear to the desired minimum, heat-treated cast-iron liners having much greater hardness are used. The same principles apply to utilization of cast iron for machine tools, or any other machinery where maximum wear resistance is desired. Typical examples of gray irons being used where resistance to wear is desired are: brake drums, passenger-car cylinder blocks, bus or truck cylinder blocks, hydraulic-press cylinders, pistons (automotive), machine-tool carriages, planer carriage and bed, brake shoes and locomotive cross-head guides, etc.

*Wear Testing.*—A number of investigators have conducted laboratory tests to determine the factors influencing the wear resistance of cast iron. Although many of those investigators used test methods not designed to simulate any particular set of conditions that might be encountered in service, the conclusions reached have undoubtedly helped us to find better materials of construction for resisting wear. Some of the conclusions drawn from laboratory tests are:

(*a*) Wear resistance increases with pearlite content(51).

(*b*) Irons possessing a pearlite matrix are most resistant to wear(53, 57).

(*c*) Wear is proportional to hardness, being less as pearlitic condition is reached(55).

(*d*) The resistance to wear increases rapidly with increasing combined carbon content from 0 up to 0.60 per cent, and then slightly from 0.60 up to 0.85 per cent(54).

(*e*) Permanent mold cast iron wears many times faster than sand-cast iron of the same combined carbon content(54).

(*f*) Wear decreases with increasing hardness with many exceptions(53).

(*g*) Wear resistance increases rapidly with hardness from 129 to 180 Brinell and remains practically constant up to 260 Brinell(57).

(*h*) Brinell hardness shows no relation to wear(51).

(*i*) Chemical composition shows no relation to wear(51).

(*j*) Increasing silicon content decreases the resistance to wear(53).

(*k*) Phosphide eutectic has a harmful influence on wear as it tends to form a grinding substance(51).

(*l*) Phosphorus in the range 0.1 to 0.75 per cent improves wear resistance(52).

(*m*) Graphite in cast iron acts as a lubricant and diminishes wear(51).

(*n*) Minimum wear is obtained when both cast irons in contact are of the same hardness (test conducted under load of 33,600 lb. per sq. in.)(55).

(*o*) Locomotive valve rings under 140 Brinell wore most rapidly. With a 10-point hardness differential between valve rings and valve box the wear was least, being only one-half that when both were the same hardness(56).

*Corrosion Resistance of Cast Iron:*

*Fields for Cast Iron*(58).—The corrosion resistance of cast iron has received much study during the past years, but the problems involved are so intricate that few comprehensive data are available.

In general, ordinary gray iron has a considerable degree of resistance to underground corrosion, as is evidenced by the fact that water pipes have given constant service for over 100 years. Cast iron is also widely used in the oil and gas fields, and in the chemical industries.

Cast iron can resist concentrated sulfuric acid (over 60 deg. Baumé), if not extremely hot; but dilute sulfuric acid attacks gray iron severely. Oleum, or fuming sulfuric, also attacks gray iron and will decompose it if the grain structure is open.

Nitric acid, which ranks next to sulfuric in commercial importance, is strongly corrosive in contact with gray iron. A high-silicon iron, or better, a 14 to 30 per cent chromium iron or steel, resists nitric acid satisfactorily.

Caustic and alkaline solutions under certain definite conditions do not attack gray iron appreciably. For this reason, cast iron is used extensively in the handling of such materials.

*Causes and Theories.*—Because of the complexity of corrosion problems, no general rules can be laid down. In the first place, corrosion may be caused by oxidation, or the chemical action of corrosive media, such as acids or alkalies, or by electrolysis, or by a combination of any two or more causes. In the second place, *every case of corrosion is different.* The temperature, the structure and composition of the iron, the agents to which it is exposed, and many other factors, influence the rate and extent of corrosion. For example, the corrosion of cast-iron water pipe is not only influenced by the nature of the pipe itself, the water flowing through it, and the soil in which it is laid, but also by possible stray electric currents.

However, work done on corrosion of iron and steel has developed certain definite facts concerning the causes of corrosion, and these will be summarized below.

From a purely chemical viewpoint, the corrosion of iron is due to the action of moisture and free oxygen to form ferrous and ferric oxide. Where the water in contact with the iron is in a closed system, as in cast-iron water mains, the free oxygen in the water usually is the principal cause of corrosion.

Since iron is soluble in acids, and to some extent in alkalies, these agents cause corrosion. Salts such as bicarbonates, chlorides, sulfates and nitrates affect iron, but free oxygen is necessary for serious damage. The failure of cast iron which has been continuously exposed to damp, oxidizing atmosphere is due to chemical action, and is often accelerated by the removal of decomposition products by steam, abrasives, solutions of acids and alkalies, and their salts(59).

Electrolysis produces much of the corrosion noted in cast iron. There are two types of electrolysis; the first is the result of an outside generated electric current flowing from the metal by a conduction path(60). This is the kind of electrolysis which attacks cast-iron pipe laid adjacent to trolley lines, etc. The second is auto-electrolysis, electrochemical in nature, the result of an electromotive force set up by the difference in potential along the surface of the metal. These differences of potential are set up by the different constituents forming couples in the surface, as graphite-iron, iron - iron oxide, etc., or by differences in oxygen content in the medium itself. In pipe lines, current has been found to flow short distances, as from a ground or chipped spot on a pipe to the adjacent original surface, and through long distances, as from a low marshy spot along the line to high ground where the oxygen supply was plentiful.

In some old cast-iron water pipes recently uncovered after over 100 years of service, graphite cemented with iron oxide was the only constituent remaining(61). Yet these pipes, when dug up, were withstanding 80 lb. pressure.

Frequently, chemical corrosion of water mains is accelerated by stray electric currents. Corrosion often results from a combination of chemical, electrolytic, and electrochemical actions.

*Iron versus Steel.*—The relative value of cast iron and steel for corrosion under natural conditions (soil and water) is well stated by Speller(62) as follows:

"Rust often adheres better to the rough surface of cast iron than to steel or wrought iron, and in atmosphere forms a fine-grained protective skin which probably accounts for the relatively longer life of cast iron in air or under wet and dry conditions. When continuously immersed, however, the rust formed has an open structure which is of little protective value.

"Several comparisons have been mentioned in connection with the tests referred to earlier in this chapter. They indicate that, in corrosive soils, both materials will suffer about the same, but that cast iron usually lasts longer on account of its greater thickness, and that, in certain cases where graphitic disintegration occurs, the pipe sometimes continues to give service for years after its strength and ductility have been materially reduced by this process. The life of cast iron and steel has been estimated to be about in proportion to the relative thickness of the metal."

Speller further quotes Friend and Marshall as follows: "Cast iron has the advantage when exposed to the air or to wet and dry tests under all conditions. In sea water, cast iron appears to be slightly more durable, but when continuously immersed in fresh water there is no material difference."

The results of the U. S. Bureau of Standards Soil Corrosion tests show that the corrosion rates of all ferrous materials are approximately the same after ten years' exposure.

As cast iron usually is furnished much thicker than steel, it is deservedly popular for water and gas mains. A further advantage is derived from the considerable strength of the graphitic residue which "plugs" holes quite effectively in severe cases of highly concentrated electrolysis or other corrosion.

*Structure.*—As between two different cast irons in the same medium, discussion of corrosion has apparently been confined to "skin effect" and grain size. Most tests have shown the casting skin to be of very little value against corrosion. What is gained by protection is lost by increased tendency to pitting by galvanic corrosion. As to grain size, it is so intimately associated with composition that it is indeed difficult to generalize. However, the work of Bardenheuer and Zeyen(63) shows that in the dilute non-oxidizing acids, hydrochloric, sulfuric and acetic, the larger the graphite particles the slower the corrosion rate *provided* the amount of graphite remains the same and all specimens are sound. This, of course, verifies the electrochemical theory of such corrosion, for the larger the graphite particles the less surface of graphite to form cells. Generally, however, in a fine-grained iron we have less graphite and consequently fewer couples. Also, there is less likelihood of porosity along the graphite flakes or between grains. Therefore, the corrosion resistance is generally better, as commonly claimed.

Tests made in natural waters show little difference due to grain size.

A low-carbon, high-silicon iron, with some such alloying element as chromium to inhibit graphitization, has been used advantageously in some plants. A low-silicon, low-phosphorus, low-sulfur iron has been developed recently for caustic work. Its composition is: silicon 0.60 to 0.75 per cent, manganese 0.15 to 0.75 per cent, phosphorus 0.08 to 0.15 per cent, total carbon 3.40 to 3.55 per cent.

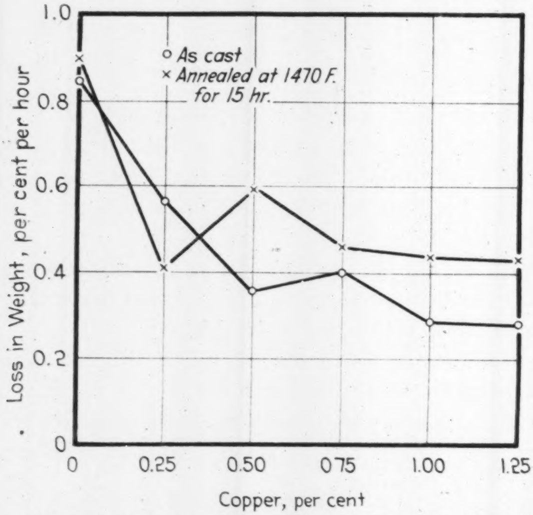

FIG. 45.—Effect of Copper on Corrosion of Cast Iron in Hydrochloric Acid Solution. (W. Denecke.)

Composition of iron: 4.15 per cent carbon, 1.29 per cent silicon, 0.26 per cent manganese, 0.004 per cent sulfur, 0.07 per cent phosphorus, copper as shown.

*Surface Defects.*—It is of the greatest importance that cast irons subjected to corrosion have a surface unbroken by defects such as slag, sand, etc., which may act as foci for corrosion. The metal must be sound and free from shrinks, pin holes and other cavities.

*Effect of Temperature.*—Corrosion problems become more acute at elevated temperatures, as nearly all chemical agents are more active when heated and therefore have more effect on the iron. Also, the growth of the castings opens cracks which offer ready access to corrosive liquids and gases. The extent of corrosion in plain water in a closed system increases rapidly with higher temperatures.

*Copper.*—Opinions differ as to the effect of copper on the corrosion resistance of cast iron. Some work indicates that copper

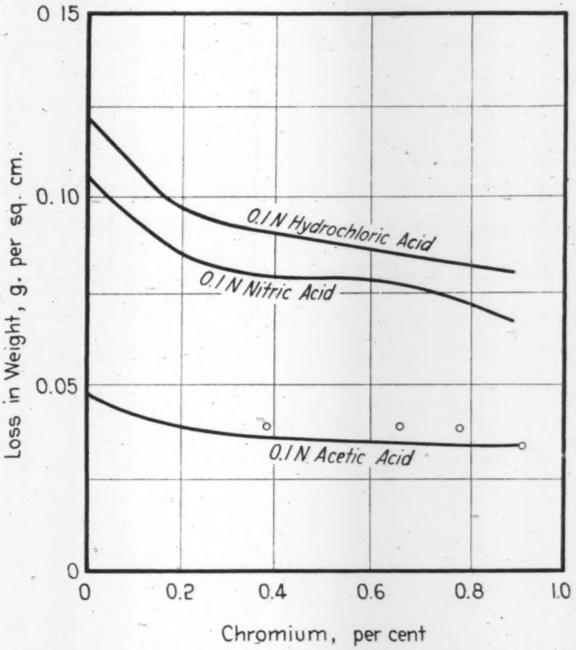

FIG. 46.—Effect of Chromium on Corrosion of Cast Iron in Various Media (J. W. Donaldson.)

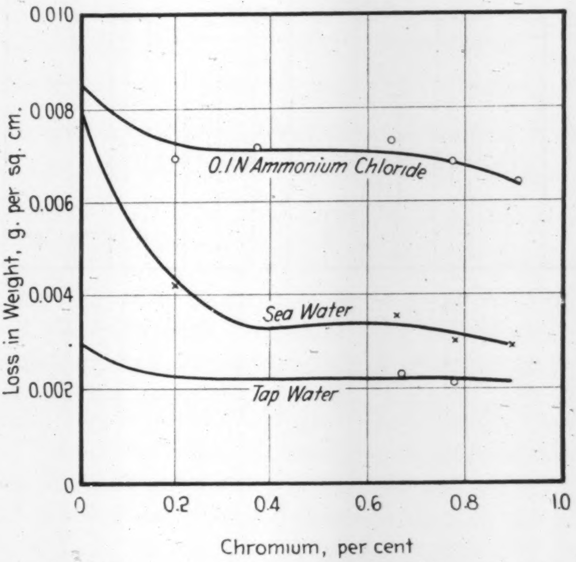

FIG. 47.—Effect of Chromium on Corrosion of Cast Iron in Various Media. (J. W. Donaldson.)

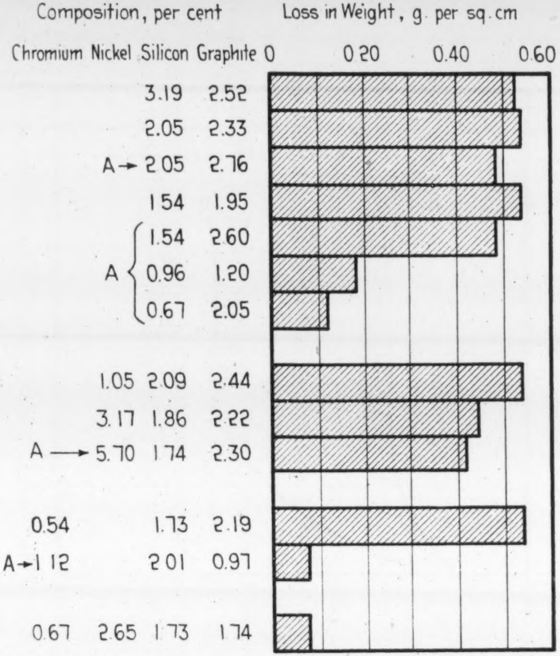

FIG. 48.—Corrosion of Various Cast Irons, After Three Days Immersion in 0.5 $N$ Hydrochloric Acid. (Koetzschke and Piwowarsky.)

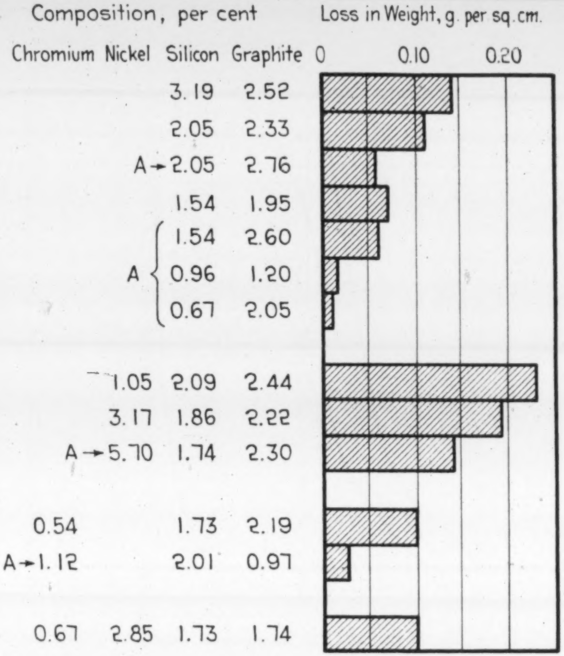

FIG. 49.—Corrosion of Some Cast Irons as Shown in Fig. 48, After Six Days Immersion in 0.2 $N$ Acid. (Koetschke and Piwowarsky.)

increases corrosion resistance in dilute acids; other work contradicts those conclusions. Experimental work, and to a greater extent, practical experience, have shown that the addition of small percentages of copper, say up to 1 per cent, have a decidedly beneficial effect in promoting corrosion resistance in certain media. Michailoff(64) states that copper is very effective in retarding corrosion of cast iron in dilute acids. Denecke(65) corroborates these results, as is shown in Fig. 45.

In practical applications cast iron containing from 0.5 to 1.0 per cent copper has been used successfully in many cases, such as pipe lines in oil refinery work. Copper-bearing cast iron is superior to plain cast iron in atmospheric corrosion.

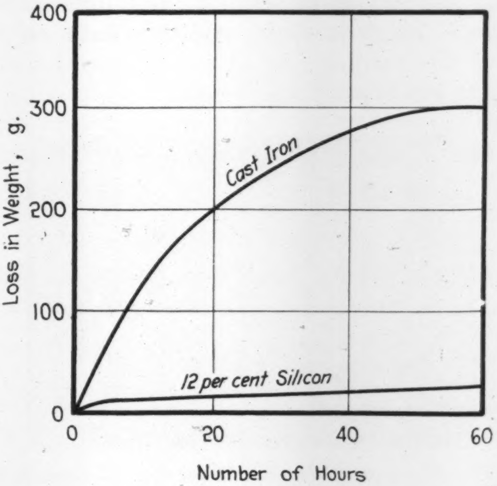

FIG. 50.—Effect of 12 per cent Silicon on Corrosion of Cast Iron in Boiling Sulfuric Acid. (W. Deneke.)

*Chromium.*—Small amounts of chromium from 0.2 to 1.0 per cent increase corrosion resistance appreciably in some media. Donaldson(66) shows in Figs. 46 and 47 that chromium is especially effective in lessening the corrosive action of sea water and weak acids.

His results are supported by the work of Koetzschke and Piwowarsky(67), who find percentages of chromium over 0.90 per cent have a marked effect on the corrosion resistance of iron. This effect, which is shown graphically in Figs. 48 and 49, is explained by the presence of the stable double carbides formed by chromium.

*Nickel.*—Small percentages of nickel apparently have little effect on the corrosion resistance of cast iron. For resistance to caustic, nickel contents from 1 to 3.5 per cent have given excellent results in practical installations.

*Nickel Chromium.*—Experience has shown that small percentages of chromium and nickel, in the usual proportions of 1.50 to 2.50 parts of nickel to 1 part of chromium, retard corrosion in some instances. Cast iron fittings containing 0.50 per cent nickel and 0.25 per cent chromium are often used, and even in these small amounts, the combination of nickel and chromium appears to increase corrosion resistance. Slightly larger amounts, from 0.60 to 1.25 per cent nickel and 0.30 to 0.60 per cent chromium, are being used successfully for the hot section of sulfur dioxide lines in acid plants(68).

*Molybdenum.*—There is little available data on the effect of molybdenum on the corrosion of cast iron, but the indications are that molybdenum has little, if any, effect.

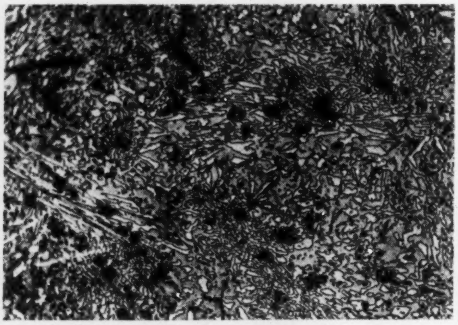

FIG. 51.—Microstructure of a 15-per-cent Chromium Iron. (Union Carbide and Carbon Research Labs.)

*Silicon.*—As stated above, close-grained irons are generally considered more resistant to corrosion than more open irons. For this reason, cast irons containing less than 0.85 per cent silicon are sometimes used under corrosive conditions. As the silicon content is increased from 1.0 to 3.0 per cent, corrosion resistance is usually lowered. (Silicon from 12 to 17 per cent is treated in another paragraph.)

*Erosion Abrasion.*—The erosive effect of continued flow of liquids and gases and abrasive particles, etc., often accelerates corrosion. Therefore, corrosion can sometimes be retarded by using iron sufficiently hard to resist abrasion and erosion. This is particularly true in oil refinery and mining castings.

*Special Corrosion-Resistant Cast Irons.*—Three classes of cast irons have been found to be particularly corrosion resistant. These are: High-silicon, high-chromium and high-nickel irons.

(*a*) *High-Silicon Iron.*—From 11 to 17 per cent silicon, cast iron acts as a corrosion-resistant alloy, being especially effective between 14.5 and 17 per cent. Irons with approximately 15 per cent silicon are used under extremely corrosive conditions with great success(69). They are remarkably good for withstanding all acids except hydrofluoric(70), and hot concentrated hydrochloric acid. This is illustrated in Fig. 50.

The disadvantage of these materials is that they are very hard and brittle, and must be ground instead of machined. They are produced under various trade names in this country and abroad. These irons find many applications in the chemical and allied industries, especially for mechanical equipment for handling corrosive liquids and gases(71).

(*b*) *High-Chromium Iron.*—When present in amounts from 15 to 30 per cent, chromium imparts a protective coating to cast iron which is especially resistant to sulfur and acid corrosion. Figure 51 shows the microstructure of a 15-per-cent chromium cast iron.

Samples containing various amounts of chromium were held for 528 hr. in a synthetic smoke atmosphere of carbon dioxide, sulfur dioxide, steam and air at 932 F., and results again indicated the formation of a corrosion-resisting coating, as follows:

| CHROMIUM | LOSS, G. PER SQ. CM. IN 528 HR. |
|---|---|
| 0 | 0.0242 |
| 11 | 0.0080 |
| 14 | 0.0071 |

Unpublished data, Union Carbide and Carbon Research Laboratories.

Some of these alloys have considerable carbon and may be classified either as cast irons or as cast steels. The composition 24 per cent chromium, 1.5 per cent silicon, and 2.00 per cent carbon is especially resistant to corrosion, although complete data are not available(72).

Cast irons containing 34.2 per cent chromium, 1.2 per cent silicon and 2.3 per cent carbon are very resistant(73). They are immune to atmospheric attack and to many chemicals as is shown in Table XXX.

The terms employed in Table XXX to denote various degrees of resistance correspond to the following losses in weight:

| LOSS IN WEIGHT, G. PER SQ. M. PER HR. | DEGREE OF RESISTANCE |
|---|---|
| Less than 0.1 | fully resistant |
| 0.1 to 1.0 | satisfactorily resistant |
| 1.0 to 3.0 | fairly resistant |
| 3.0 to 10.0 | slightly resistant |
| Over 10.0 | not resistant |

TABLE XXX.—RESISTANCE OF HIGH-CHROMIUM IRONS TO CHEMICAL ATTACK.
(*Metals and Alloys*, February, 1933.)

| MEDIUM OF ATTACK | TEMPERATURE | DEGREE OF RESISTANCE | |
|---|---|---|---|
| | | 1.1 PER CENT CARBON | 2.3 PER CENT CARBON |
| Nitric Acid: | | | |
| 7 per cent, specific gravity 1.04 | 20 C. (68 F.) | fully | fully |
| 7 per cent, specific gravity 1.04 | boiling | fully | fully |
| 37 per cent, specific gravity 1.23 | 20 C. (68 F.) | fully | fully |
| 37 per cent, specific gravity 1.23 | boiling | satisfactory | satisfactory |
| 66 per cent, specific gravity 1.40 | 20 C. (68 F.) | fully | fully |
| 66 per cent, specific gravity 1.40 | boiling | satisfactory | satisfactory |
| concentrated fuming, specific gravity 1.52 | 20 C. (68 F.) | satisfactory | satisfactory |
| concentrated fuming, specific gravity 1.52 | 40 C. (104 F.) | not resistant | not resistant |
| Mixed Acid, per cent by weight: | | | |
| 30 per cent concentrated sulfuric acid plus 5 per cent concentrated nitric acid plus 65 per cent water | 50 C. (122 F.) | fully | fully |
| Sulfurous Acid, saturated aqueous solution | 20 C. (68 F.) | fully | satisfactory |
| Phosphoric Acid: | | | |
| 10 per cent, specific gravity 1.05 | boiling | fully | fully |
| 45 per cent, specific gravity 1.30 | boiling | fully | fully |
| 80 per cent, specific gravity 1.64 | boiling | slightly | slightly |
| Acetic Acid: | | | |
| 10 per cent, specific gravity 1.01 | boiling | fully | fully |
| 80 per cent, specific gravity 1.07 | boiling | fully | fully |
| 100 per cent, specific gravity 1.05 | boiling | fully | fully |
| Lactic Acid, 1.5 per cent | boiling | fully | fully |
| Sodium Hydroxide: | | | |
| Aqueous solution, 15 per cent, specific gravity 1.16 | 50 C. (122 F.) | fully | fully |
| Aqueous solution, 15 per cent, specific gravity 1.16 | boiling | fully | fully |
| 50 per cent, specific gravity 1.53 | boiling | not resistant | not resistant |

TABLE XXX.—*Continued.*

| Medium of Attack | Temperature | Degree of Resistance<br>1.1 per cent Carbon | Degree of Resistance<br>2.3 per cent Carbon |
|---|---|---|---|
| Ammonium Nitrate, aqueous solution 50 per cent...... | boiling | fully | fully |
| Ammonium Sulfate, aqueous solution 50 per cent...... | boiling | fully | fully |
| Calcium Bisulfite: | | | |
| at 12 to 15 atmospheres pressure.................... | about 150 C. (300 F.) | not resistant | not resistant |
| at 4 to 6 atmospheres pressure.................... | about 90 C. (194 F.) | fully | fully |
| Calcium Hypochlorite, aqueous solution saturated cold | 40 C. (104 F.) | satisfactory | satisfactory |
| Ferric Chloride, aqueous solution 30 per cent......... | 20 C. (68 F.) | not resistant | not resistant |
| Potassium Nitrate, aqueous solution 25 per cent...... | boiling | fully | fully |
| Sodium Sulfite, aqueous solution 50 per cent......... | boiling | fully | fully |
| Zinc Chloride, aqueous solution: | | | |
| Specific gravity 1.20....... | boiling | fully | fully |
| Specific gravity 1.90....... | boiling | not resistant | not resistant |

TABLE XXXI.—RATES OF CORROSION OF NICKEL CAST IRON IN 130° TWADDELL CAUSTIC AT THE BOILING POINT.

| | Total Carbon, per cent | Silicon, per cent | Nickel, per cent | Chromium, per cent | Loss in Weight, mg. per sq. dm. per Day |
|---|---|---|---|---|---|
| No. 1........................ | 3.66 | 0.97 | none | none | 366 |
| No. 2........................ | 3.03 | 1.11 | 3.77 | none | 235 |
| No. 3........................ | 2.88 | 0.75 | 6.53 | none | 247 |
| No. 4........................ | 2.73 | 0.60 | 16.66 | none | 152 |
| No. 5........................ | 2.67 | 0.65 | 19.55 | 1.80 | 31 |
| No. 6........................ | 2.13 | 0.51 | 22.24 | none | 17 |
| No. 7........................ | 2.27 | 0.61 | 28.90 | none | 2 |

RECOMMENDED COMPOSITIONS, PER CENT

| | Total Carbon | Silicon | Nickel | Chromium | Copper |
|---|---|---|---|---|---|
| Copper-free Ni-Resist..... | 2.80 to 3.10 | 0.80 to 1.40 | 18 to 22 | 2 to 4 | ...... |
| Regular Ni-Resist........ | 2.80 to 3.10 | 1.25 to 2.00 | 12 to 15 | 2 to 3.5 | 5 to 7 |

TABLE XXXII.—COMPARATIVE RATES OF CORROSION (International Nickel Co.).

| Reagent | Austenitic Cast Iron | Ordinary Cast Iron | Brass | Bronze |
|---|---|---|---|---|
| 5 per cent sulfuric acid cold............ | 1 | 475 | 1.12 | 1.08 |
| 10 per cent sulfuric acid at 100 C. (212 F.)........................... | 1 | 195 | 0.15 | 0.15 |
| 78 per cent sulfuric acid at 100 C. (212 F.)........................... | 1 | 1.95 | ..... | 0.08 |
| 5 per cent hydrochloric acid............ | 1 | 185 | 13.0 | 24.0 |
| 25 per cent acetic acid................... | 1 | 400 | 2.75 | 2.22 |
| Salt spray.............................. | 1 | 2.9 | 0.27 | 0.08 |
| Sea water............................... | 1 | 5.0 | 0.5 | 2.15 |

(*c*) *High-Nickel Iron.*—High-nickel cast irons are austenitic in structure and extremely corrosion resistant. They have the added advantage of easy machineability. A cast iron having the approximate composition of 15 per cent nickel, 2.5 per cent chromium, 6 per cent copper and 1 per cent manganese has high resistance to many acids and alkalies. It is recommended for hydrochloric, sulfuric and phosphoric acids, but also gives good results with caustic solutions. Where a low corrosion rate is desired or freedom from metal contamination is necessary, cast irons containing up to 20 per cent nickel are commonly used. See Table XXXI.

TABLE XXXIII.—CORROSION TESTS (International Nickel Co.).

| | WEIGHT LOSS, MG. PER SQ. DM. PER DAY | | |
|---|---|---|---|
| REAGENT | NI-RESIST CAST IRON | ORDINARY CAST IRON | RATIO, ORDINARY CAST IRON TO NI-RESIST CAST IRON |
| Atmosphere | Rusts superficially | Rusts badly | .... |
| Atmosphere 30 days | 9.5 | 59.7 | 6 |
| Atmosphere 90 days | 7.9 | 63.5 | 8 |
| Atmosphere 1½ years | 3.5 | 35.0 | 10 |
| Water spray, vertical | 6.6 | 207.5 | 32 |
| Water spray, horizontal | 17.6 | 244 | 14 |
| Aerated tap water | 7.8 | 67.2 | 9 |
| 3 per cent salt solution | 50.0 | 190 | 4 |
| 12½ per cent fermented molasses solution | 10.0 | 360 | 36 |
| Bad boiler water 95 C. (203 F.) | 110 | 660 | 6 |
| Ferric sulfate | 17 000 | 32 000 | 2 |
| 5 per cent sulfuric acid aerated | 350 | 30 000 | 85 |
| 5 per cent hydrochloric acid aerated | 507 | 26 665 | 50 |
| 10 per cent hydrochloric acid aerated | 598 | 29 475 | 50 |
| 20 per cent hydrochloric acid aerated | 1 111 | 33 270 | 30 |
| Hot caustic | 30.0 | 430 | 8 |

For use in bleaching plants, a copper-free austenitic cast iron has been developed with the following composition: Total carbon 2.70 to 3.10 per cent, silicon 0.80 to 1.50 per cent, nickel 18 to 22 per cent, chromium 2 to 3.50 per cent. This iron is also recommended for use in caustic work where freedom from copper contamination is desired. Figure 52 illustrates the difference in corrosion resistance between ordinary cast iron and the austenitic variety.

Tables XXXII and XXXIII serve to illustrate the excellent corrosion resistance of the austenitic cast irons.

*Heat Resistance of Cast Iron:*

The two conditions which cast irons must overcome for satisfactory high temperature service are growth and oxidation.

*Growth of Gray Iron*(74).—The growth of gray iron is exhaustively treated in an article by J. W. Bolton and Hyman Bornstein(74), and this material as it applies to ordinary cast iron has been abstracted in the section on Growth of Iron.[1]

Ordinary cast irons are serviceable at temperatures below 650 F. but growth begins above this temperature. Moreover, cast irons which give excellent service under usual conditions exhibit growth under 650 F. in the presence of superheated, high-pressure steam. Above 650 to 700 F., irons grow not only as the result of alternate expansion and contraction, and the penetration of corrosive gases, but also as the result of graphitization. Especially above 900 F. the decomposition of pearlite into ferrite and graphite increases the volume and causes the rate of growth to increase accordingly. Above 1350 F. the growth is much more rapid due to the transformation of the pearlite-ferrite mixture into austenite and the intensified action of the other factors promoting growth. Castings have been known to grow as much as 50 per cent of their original volume, and such castings are not only misshapen but also extremely brittle as shown in Fig. 53.

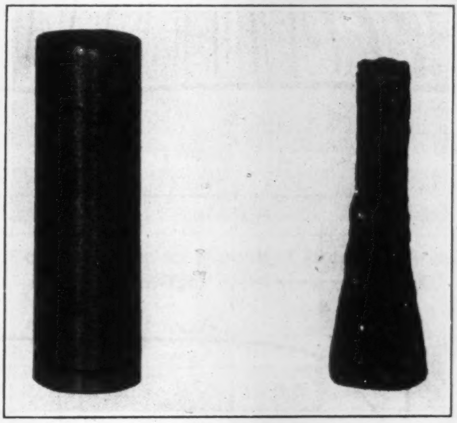

FIG. 52.—Austenitic Cast Iron (Left) and Plain Cast Iron After Exposure to Corrosive Media. (International Nickel Co.)

Alloy heat-resistant cast irons have been developed to withstand operating temperatures up to 1800 F.

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[1] See p. 203.

*Theories on Growth of Gray Iron.*—Out of the mass of work on the subject of growth, two general theories have arisen. The first

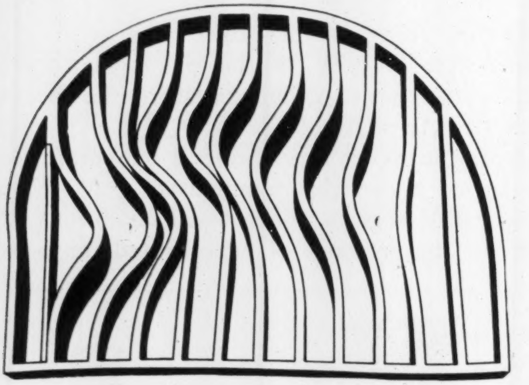

FIG. 53.—Distortion in Firebar Grating Due to Growth After Prolonged Heating. (J. E. Hurst.)

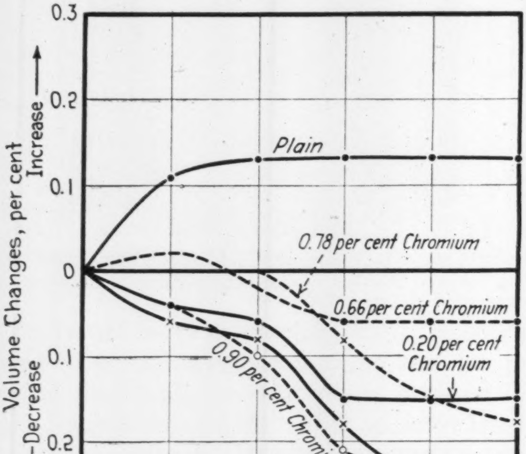

FIG. 54.—Effect of Chromium on Growth at 1020 F. (J. W. Donaldson.)

holds that growth is due to chemical action forming more bulky compounds than those originally present. Rugan and Carpenter(75, 76) believe that the growth of cast iron is primarily due to the penetration of furnace gases into the interior of the metal by way of graphite flakes, and the consequent internal oxidation of the constituents, especially silicon. The products of the oxidation of iron silicide, which are ferric oxide and silica, are both extremely bulky, and this explains a large increase in the volume of the iron.

Donaldson(77) advances the opinion that growth is caused first by decomposition of carbides and later by internal oxidation. The second theory is that growth is a mechanical phenomenon, the result of tiny fissures caused by partially irreversible expansions. Kikuta(78), Benedicks and Lofquist(79), and other noted scientists support this contention.

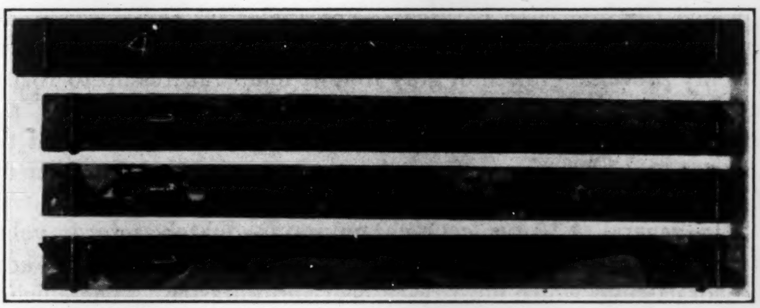

FIG. 55.—Effect of Chromium on Growth of Iron Bars After Repeated Heatings to 1650 F. (Sherwin and Kiley.)

| | MIX NO. 4[a] | MIX NO. 1 PLUS 5 PER CENT FERRO-CHROMIUM[b] | | |
|---|---|---|---|---|
| Initial bar length, in. | 11.8885 | 11.8000 | 11.8000 | 11.8000 |
| 10 heats at 1650 F., bar length, in. | 12.1870 | 11.8350 | 11.8350 | 11.8600 |
| 15 heats at 1650 F., bar length, in. | 12.3437 | 11.8350 | 11.8550 | 11.8600 |
| Increase after 10 heats, in. | 0.2985 | 0.0350 | 0.0350 | 0.0600 |
| Increase after 15 heats, in. | 0.4552 | 0.0550 | 0.0550 | 0.0600 |

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[a] Analysis of mix No. 4: Silicon 2.34 per cent, sulfur 0.09 per cent, manganese 0.63 per cent, phosphorus 0.38 per cent, total carbon 3.57 per cent, combined carbon 0.42 per cent.

[b] Analysis of mix No. 1 when 5 per cent ferro-chromium was added: Silicon 2.10 per cent, sulfur 0.10 per cent, manganese 0.67 per cent, phosphorus 0.18 per cent, total carbon 3.33 per cent, combined carbon 2.98 per cent, chromium 3.28 per cent.

*Effect of Structure and Composition.*—Whatever the explanation, it is an accepted fact that fine-grained irons with more stable carbides are much more resistant to growth than those with coarse structure. Strong irons with low total carbon are therefore more useful for high-temperature service than those with softer properties. Low-silicon or alloyed white iron is more resistant to growth than gray iron because it is harder to graphitize.

*Effect of Chromium on Growth.*—Since the irons with more stable carbides do not tend to grow, materials which stabilize carbides will retard growth. For this reason, chromium is much used to produce heat-resistant irons, since it has a very powerful stabilizing action on the carbides. It also decreases the loss of strength which usually accompanies high-temperature service.

Although any addition of chromium will retard growth to some extent, it is important to remember that the optimum amount of chromium for any given result depends on the carbide-forming characteristics of the iron to be treated. For a low-silicon, low-total-carbon iron, less chromium is necessary than for a softer iron. The amount of chromium needed also varies with the temperature at which the iron is to be used. Researches by Donaldson(80) indicate that with certain irons as little as 0.20 to 0.39 per cent chromium not only prevents growth of gray iron at temperatures up to 1020 F., but actually causes the metal to show a slight contraction, as is evident in Fig. 54. More recent work(81) has shown that less than 0.80 per cent chromium effectively prevents growth in another type of iron, up to 1110 F., as illustrated in Fig. 16.

In general, 1.50 per cent chromium adequately retards volume changes up to 1600 F.(82). However, slightly more chromium is advisable for castings which must resist heat and abrasion. The efficiency of 3.28 per cent chromium iron in this regard is shown(83) in Fig. 55.

At temperatures up to 1800 F., castings must withstand both heat and oxidation. Therefore, from 14 to 34 per cent chromium is required at such temperatures.

A cast iron containing 24 per cent chromium shows practically no growth, and gives high resistance to oxidation. This iron usually is made in the electric furnace, with the following composition: chromium 24 per cent, silicon 1.50 per cent, carbon 2.00 per cent.

Valenta(84) states that an iron containing 1.43 per cent carbon, 2.30 per cent silicon, and 27.91 per cent chromium, was heated for 24 hr. at 1470 F. for comparison with an ordinary cast iron. The latter grew in length 1.243 per cent, while the chromium cast iron exhibited no measurable growth within the limits of experimental accuracy. Valenta brings out the important point that "such alloys without losing their heat-resisting properties become more ductile and resistant to shocks."

Cast irons containing 34 per cent chromium have recently been investigated and found to show no growth on heating. They possess great resistance to scaling and are immune from oxidation up to temperatures as high as 2010 F. Moreover, the thermal expansion is regular because this iron undergoes no transformation and even frequent changes of temperatures do not cause deformation of castings(85).

*Effect of Silicon on Growth.*—In the amounts ordinarily found in cast iron, silicon increases graphitization and therefore growth. However, very small or very large amounts of silicon retard growth.

A low-silicon iron called "Perlit" is to some extent heat resistant due to its completely pearlitic structure and fine, uniformly distributed graphite. A peculiarity of this iron is that if it were cooled normally, it would solidify as a white iron, but when cooled in preheated molds, it produces a gray iron. Its composition is approximately 3.25 per cent total carbon, 1.11 per cent silicon, 0.79 per cent manganese, 0.40 per cent phosphorus, 0.15 per cent sulfur. A harder mixture (2.9 per cent carbon, 0.9 per cent silicon) is used for thick sections. Besides its resistance to heat and oxidation, this metal possesses high strength, resistance to impact, machineability and moderate hardness.

TABLE XXXIV.—GROWTH OF CAST IRON (BRITISH CAST IRON RESEARCH ASSOCIATION).

| COMPOSITION | | |
|---|---|---|
| CARBON, PER CENT | SILICON, PER CENT | GROWTH, PER CENT |
| 2.1 | 3.7 | 0.923 |
| 2.1 | 5.3 | 0.571 |
| 1.95 | 5.6 | 0.247 |
| 2.5 | 6.1 | 0.011 |

TABLE XXXV.—EFFECT OF NICKEL AND CHROMIUM ON CARBIDE STABILITY.

| | | | | | COMBINED CARBON, PER CENT | | |
|---|---|---|---|---|---|---|---|
| | TOTAL CARBON, PER CENT | SILICON, PER CENT | NICKEL, PER CENT | CHROMIUM, PER CENT | AS CAST | AFTER 90 HR. AT 1200 F. | PERCENTAGE OF ORIGINAL |
| Plain | 3.10 | 1.69 | .... | .... | 0.75 | 0.09 | 11.5 |
| Nickel-Chromium | 3.15 | 1.76 | 1.13 | 0.45 | 0.90 | 0.41 | 45.5 |

This pearlitic structure may be obtained by casting at very high temperatures, without preheating the molds.

A cast iron with 6 to 8 per cent silicon has recently been developed by the British Cast Iron Research Association(86). The resistance of this metal to growth depends upon: (1) an entirely ferritic structure, which prevents carbide decomposition; (2) alloy additions which raise the transformation temperature above the working range, thus avoiding open structure and consequent internal oxidation resulting from volume changes; (3) fine-grained, "supercooled," graphite; (4) mechanically dense metallic matrix.

Table XXXIV shows the growth of various cast irons after ten heatings at temperatures up to 1830 F.

This iron is recommended for service at 1300 to 1650 F. but when the stresses are likely to be applied in the cold state, it is advisable to use an iron with similar percentages of silicon, but with the addition of other alloys to improve ductility. These alloyed high-silicon irons are mentioned below.

Effect of heating various steels and cast irons at 1830 F., for seven half-hour periods is shown in Fig. 56.

*Effect of Nickel and Chromium on Growth.*—Castings showing considerable resistance to growth can be made by adding enough chromium to stabilize the carbides and enough nickel to insure machineability. A typical composition for such an iron is nickel 1.50 per cent, chromium 1.00 per cent.

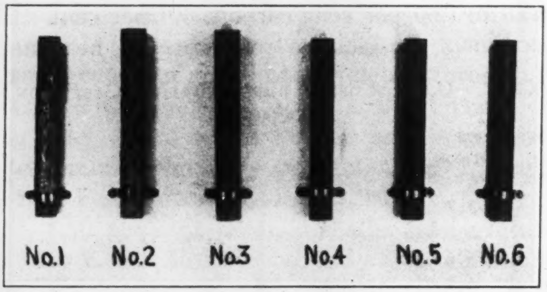


FIG. 56.—Test Bars Showing Condition of Steel and Various Cast Irons After Seven Half-Hour Periods of Heating at 1000 C. (1830 F.). The following results were obtained, the test bars being numbered from left to right:

No. 1. **Mild steel.** No growth but excessive scaling.

No. 2. **Engineering** cast iron (1.7 per cent silicon). **Severe scaling; growth 16** per cent.

No. 3. **Cast iron** (3.7 per cent silicon). Very little scaling; **growth 12** per cent.

No. 4. "**Silal**" cast iron (5.6 per cent silicon). **Slight surface oxidation;** growth 5.0 per cent.

No. 5. "**Silal**" **cast** iron (7.5 per cent silicon). **Very slight surface** oxidation; growth 2.7 per cent.

No. 6. "Nicrosilal" cast iron (6 per cent silicon, 17 per cent nickel, 3 per cent chromium). Surface practically untarnished; growth 1.3 per cent.

The change in combined carbon as shown in Table XXXV is a measure of the decomposition of the carbide, and therefore the growth of the cast irons(**87**).

Striking new developments for high temperatures are austenitic cast irons with the following compositions:

| | Nickel, per cent | Silicon, per cent | Chromium, per cent | Manganese, per cent | Copper, per cent |
|---|---|---|---|---|---|
| Iron No. 1 (**88**) | 14 | 2 | 4 | 1.25 | 6 |
| Iron No. 2 (**89**) | 18 | 6 | 2 | 1.00 | .... |

Because they are already austenitic, these irons do not undergo a structural transformation and subsequent volume changes on repeated heating and are therefore more resistant to growth than is plain cast iron. They withstand oxidation, especially at temperatures between 1200 to 1500 F., as shown in Fig. 57.

Figure 58 shows the relative growth, oxidation and warping of plain gray iron and such an alloy iron after repeated heatings. These alloys can be made in the cupola, electric furnace, or crucible. Because of their heat-resisting qualities, and the ease with which they are molded, cast, machined, and welded, these austenitic irons have found many uses in such castings as valves, fittings, boiler specialties, propellers, stove and furnace parts, etc.

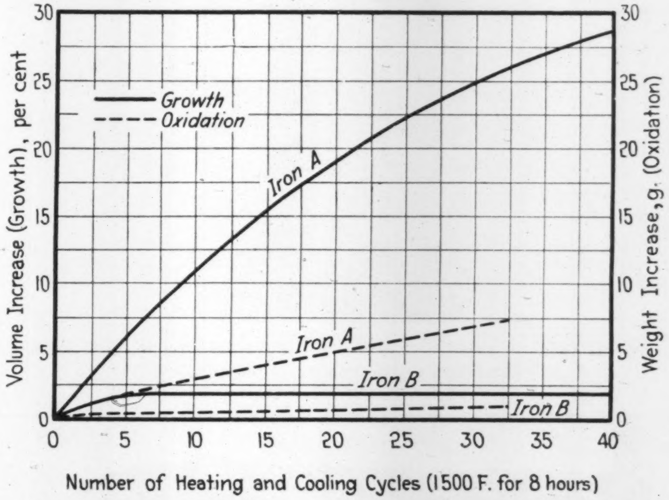


FIG. 57.—Curve Showing Oxidation and Growth of Plain Cast Iron (*A*) and Austenitic Cast Iron (*B*). (International Nickel Co.)

| | CHEMICAL ANALYSIS, PER CENT | |
|---|---|---|
| | IRON A | IRON B |
| Total carbon | 3.26 | 3.04 |
| Silicon | 2.02 | 1.62 |
| Nickel | .... | 14.31 |
| Copper | .... | 5.37 |
| Chromium | .... | 3.23 |

*Effect of Molybdenum, Tungsten, Vanadium, and Manganese on Growth.*—Fundamentally, it is safe to say that any alloy which tends to stabilize the carbides will promote heat resistance. Therefore, it is highly probable that tungsten, molybdenum, vanadium, and manganese have a beneficial effect in improving the heat resistance of cast iron, although conclusive data are as yet lacking.

*Fire Cracks.*—In cases where only the surface of the cast iron is exposed to temperatures above red heat, a slight permanent growth takes place on the surface. On cooling and repeated heating, small cracks, known as "fire cracks," appear. (See Fig. 59.) These fire cracks, besides weakening the iron, allow easy ingress of gases and other corrosive media. They eventually cause the destruction of the cast iron, particularly if in hard service, such as in heavy duty brake drums, metal molds, etc. It has been found that these cracks are

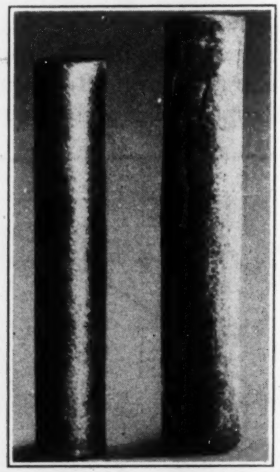


FIG. 58.—Comparison of Austenitic (Left) to Plain Cast Iron (Right) After Repeated Heatings. (International Nickel Co.)

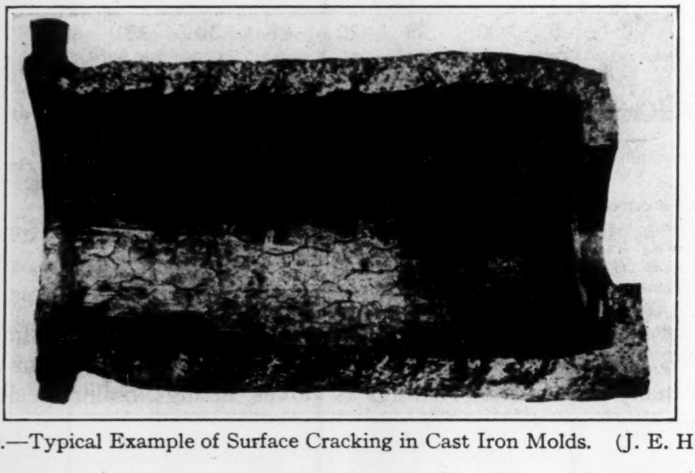


FIG. 59.—Typical Example of Surface Cracking in Cast Iron Molds. (J. E. Hurst.)

usually due to the decomposition of carbides and that the addition of a carbide stabilizer, such as chromium or molybdenum, will retard them.

*Effect of Temperature on Strength.*—Cast irons become weaker at elevated temperatures, and in most cases a permanent loss of strength results. Such weakening is quite serious and excludes ordinary cast iron from service at high temperatures. Strong, close-grained irons will minimize the weakening effect of high temperatures, but for retention of strength, chromium or molybdenum alloys, with or without nickel, are used. The effect is shown(90) in Fig. 60. The analyses of the irons of Fig. 60 are given below:

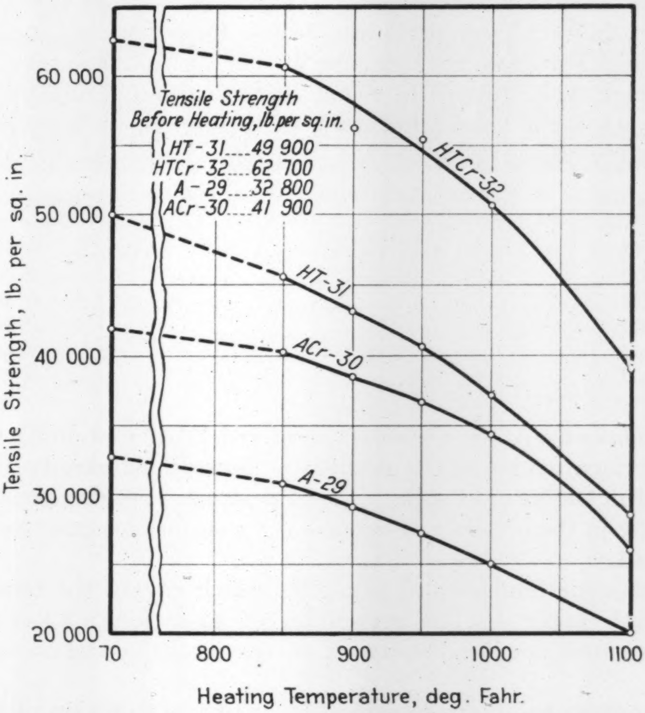


FIG. 60.—Strength of Cast Irons at Elevated Temperatures. (MacPherran and Kreuger.)

| SYMBOL | TOTAL CARBON, PER CENT | COMBINED CARBON, PER CENT | GRAPHITIC CARBON, PER CENT | SILICON, PER CENT | SULFUR, PER CENT | PHOSPHORUS, PER CENT | MANGANESE, PER CENT | NICKEL, PER CENT | CHROMIUM, PER CENT |
|---|---|---|---|---|---|---|---|---|---|
| H T-31.... | 2.96 | 0.73 | 2.23 | 2.53 | 0.113 | 0.06 | 0.78 | 0.96 | 0.02 |
| H T Cr-32. | 2.67 | 0.79 | 1.88 | 2.39 | 0.090 | 0.07 | 0.67 | 0.76 | 0.45 |
| A-29....... | 3.63 | 0.73 | 2.90 | 1.15 | 0.091 | 0.17 | 0.54 | 0.03 | 0.03 |
| A Cr-30.... | 3.46 | 0.87 | 2.59 | 1.36 | 0.085 | 0.39 | 0.68 | 0.01 | 0.54 |

The cast iron containing 24 per cent chromium cited on page 230 (25th line) exhibits very high strength at elevated temperatures. One test showed a tensile strength of 14,700 lb. per sq. in. at 1600 F. The 34 per cent chromium irons already mentioned actually increase in tensile strength from room temperature up to a point which has not been accurately determined. As is shown below, at room temperature the tensile strength was 58,500 lb. per sq. in., at 932 F., the strength had risen to 65,600 lb. per sq. in., and at 1112 F. the strength had dropped to 40,500 lb. per sq. in. From this it can be seen that the maximum strength is somewhere between room temperature and 1112 F.

| Temperature, deg Fahr. | Tensile Strength, lb. per sq. in. |
|---|---|
| 68 | 58 500 |
| 932 | 65 600 |
| 1112 | 40 500 |
| 1472 | 15 500 |
| 1652 | 9 400 |
| 1832 | 6 400 |

*Oxidation.*—At temperatures over red heat, cast irons tend to form surface oxides which eventually become sufficiently thick to drop off in the form of scale. Rapid destruction of the irons follow, especially in the presence of excess oxygen or furnace gases, as shown in Fig. 61.

The same fundamental principles which govern the retardation of growth apply to the prevention of oxidation. Close-grained structure and small additions of alloys retard oxidation to some extent.

For great resistance to oxidation, at temperatures up to 1800 F., the nickel-copper-chromium alloys cited above are often used. A new development in oxidation-resistant material is a cast iron with 15 to 40 per cent chromium. Irons containing 24 or 34 per cent chromium, already mentioned, are especially effective in retarding oxidation.

*Some Applications.*—An illustration of resistance to high temperature is shown in Fig. 62.

These new heat-resistant alloyed cast irons are finding greatly broadened fields of application, particularly in such service as grate bars, annealing boxes, manifolds, furnace parts, and stoker parts. The increased cost of such castings usually is more than compensated by lengthened service, lessened replacement and lowered maintenance costs.

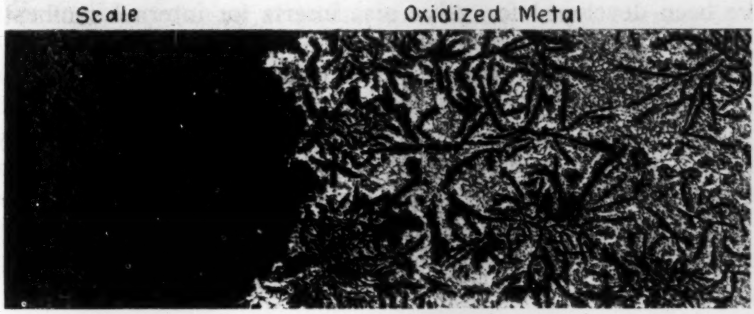


FIG. 61.—Common Iron Furnace Plate After Three Firings at 1470 F., Etched (× 75). Reduced ⅓ in reproduction. (Norbury and Morgan.)

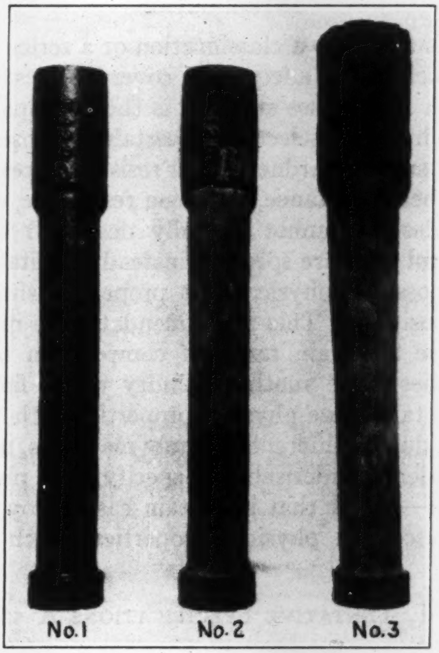


FIG. 62.—Growth of 16 per cent Chromium Cast Iron (No. 2) After Several Months Service at High Temperature as Compared with Ordinary Hard Iron (No. 3) Which Had the Same Service. No. 1 is the alloyed iron casting before being put in service. (Electro Metallurgical Co.)

Special alloy cast irons which will withstand high temperatures have been developed for valve seat inserts for internal combustion engines. One type, which is a machineable iron, has 1.00 to 1.20 per cent molybdenum with or without small additions of chromium and nickel in a cylinder block type of iron. This iron has a Brinell hardness range of 250 to 270. A second type is a more highly alloyed cast iron which has higher heat resisting properties and combines good toughness and high "red-hardness." It is white as cast in sand with a Rockwell, "C" scale, hardness of about 62. There is little loss in hardness after service in a motor. The iron is of the following type:

| | | | |
|---|---|---|---|
| Carbon, per cent | 2 | Chromium, per cent | 3.00 |
| Silicon, per cent | 1.25 | Molybdenum or tungsten, per cent | 4.50 to 5.50 |

## IV. CLASSIFICATION AND SPECIFICATIONS

### INTRODUCTION

It is difficult to set up a classification or a series of classifications for cast iron which would adequately cover the desired properties in the castings. In some cases strength is the principal factor while in many cases cast irons are selected for certain purposes on the basis of other properties, such as hardness, wear resistance, resistance to shock, machineability, heat resistance, corrosion resistance, etc. Frequently the properties desired cannot be fully described in terms of test results and control tests are specified instead of suitability tests.

Wherever possible, physical test properties should be specified instead of composition. This recommendation is made because one foundry may use a certain range of composition to secure certain physical properties while another foundry would find it much more convenient to obtain these physical properties with a different range of composition, due to differences in raw materials, melting practices, etc. It is considered undesirable to specify both physical properties and composition—except that in certain cases alloys are specified in order to obtain certain physical properties which are not readily measurable.

### A.S.T.M. TENTATIVE SPECIFICATIONS A 48 – 32 T[1]

*Tensile Strength Classification:*

The A.S.T.M. recently has issued Tentative Specifications for Gray-Iron Castings (A 48 – 32 T), which apply to gray-iron castings where strength is a consideration. In these specifications, gray-iron castings are classed according to minimum tensile strengths of test bars as shown below:

---

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 625 (1932); 1932 Book of A.S.T.M. Tentative Standards, p. 179.

| CLASS | TENSILE STRENGTH, MIN., LB. PER SQ. IN. |
|---|---|
| No. 20 | 20 000 |
| No. 25 | 25 000 |
| No. 30 | 30 000 |
| No. 35 | 35 000 |
| No. 40 | 40 000 |
| No. 50 | 50 000 |
| No. 60 | 60 000 |

*Transverse Strengths:*

In these specifications transverse tests are optional and the minimum breaking loads are specified as follows:

| | LOAD AT CENTER, LB. | | |
|---|---|---|---|
| CLASS | 0.875-IN. DIAMETER BAR, 12-IN. SUPPORTS | 1.2-IN. DIAMETER BAR, 18-IN. SUPPORTS | 2.0-IN. DIAMETER BAR, 24-IN. SUPPORTS |
| No. 20 | 900 | 1700 | 4 500 |
| No. 25 | 1050 | 1900 | 5 500 |
| No. 30 | 1200 | 2100 | 6 500 |
| No. 35 | 1300 | 2300 | 7 500 |
| No. 40 | 1400 | 2500 | 9 000 |
| No. 50 | 1700 | 2800 | 11 000 |
| No. 60 | 2000 | 3100 | 13 000 |

By agreement between manufacturer and purchaser, deflection values in the transverse test may be specified.

*Size of Test Bars:*

Three sizes of test bars are specified in order to represent various thicknesses of castings. With the manufacturer and purchaser agreeing on the controlling section of the casting, the corresponding test bar shall be used as given below:

| CONTROLLING SECTION OF CASTING, IN. | TEST BAR | NOMINAL DIAMETER OF TEST BAR AS CAST, IN. |
|---|---|---|
| 0.75, max | *A* | 0.875 |
| 0.76 to 1.10 | *B* | 1.20 |
| 1.11 to 2.00 | *C* | 2.00 |

For sections of castings over 2.00 in., test bar *C* may be used or a larger test bar may be chosen by agreement between the manufacturer and the purchaser.

The transverse test bars used have dimensions as follows:

| TEST BAR | DIAMETER, IN. | LENGTH, IN. | DISTANCE BETWEEN SUPPORTS IN TRANSVERSE TEST, IN. |
|---|---|---|---|
| *A* | 0.875 | 15 | 12 |
| *B* | 1.20 | 21 | 18 |
| *C* | 2.00 | 27 | 24 |

*General:*

There is no definite factor for correlation between tensile and transverse strengths for various types of cast iron. A table included in the specification gives minimum values only.

It is the intent of these specifications to subordinate chemical composition to physical properties. By agreement between the manufacturer and purchaser, the quantities of any chemical elements in cast iron may be specified.

### OTHER A.S.T.M. SPECIFICATIONS

The A.S.T.M. has developed other specifications for specific gray-iron castings as shown in Table XXXVI.

### HARDNESS

The hardness of a casting frequently is used as a guide to strength, machineability or wear resistance. However, it is dangerous to use hardness alone as a means of classification of castings.

### STRUCTURE

The structure of a casting as shown under the microscope gives a good indication of physical properties. Care should be taken in the selection of samples to represent the casting. It is possible that, in the future, classifications of cast iron may be made on the basis of structure.

## V. HEAT TREATMENT

The heat treatment of cast iron, when properly done and when the correct grade of cast iron is used, increases the usefulness and enhances the physical properties of the resultant product(91–104). Cast iron is directly comparable with steel in its reaction to heat treatment, the only fundamental difference in the composition of the two materials being the presence of free graphite particles in gray cast iron.

The heat treatment of cast iron may be broadly divided into three general classifications, namely, (*a*) annealing, (*b*) quenching and drawing, and (*c*) special heat treatments.

## ANNEALING

*Relieving Internal Stresses:*

The heat treatment resulting in the relieving of internal stresses is commercially called "aging," "normalizing" and "mild annealing." The relief of internal stresses in castings may be accomplished by heating to from 800 to 950 F., holding at temperature from 30 min. to 5 hr., depending on section, and cooling slowly in the furnace. This treatment will cause only slight decrease in hardness, very little decomposition of cementite and will affect inherent strength properties of the metal only slightly.

TABLE XXXVI.—A.S.T.M. SPECIFICATIONS FOR GRAY-IRON CASTINGS.

| A.S.T.M. DESIGNATION | TITLE OF SPECIFICATION | REFERENCE |
|---|---|---|
| A 44 – 04 | Standard Specifications for Cast-Iron Pipe and Special Castings | 1930 Book of A.S.T.M. Standards, Part I, p. 468. |
| A 45 – 14 | Standard Specifications for Cast-Iron Locomotive Cylinders | 1930 Book of A.S.T.M. Standards, Part I, p. 481. |
| A 46 – 30 T | Tentative Specifications for Chilled-Tread Cast-Iron Wheels | *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 976 (1930); also 1932 Book of A.S.T.M. Tentative Standards, p. 172. |
| A 126 – 30 | Standard Specifications for Gray-Iron Castings for Valves, Flanges and Pipe Fittings | 1930 Book of A.S.T.M. Standards, Part I, p. 499. |
| A 142 – 32 T | Tentative Specifications for Cast-Iron Culvert Pipe | *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 619 (1932); also 1932 Book of A.S.T.M. Tentative Standards, p. 166. |

Increasing time at temperature and higher temperatures progressively cause decrease in strength and hardness values of the metal. Soft iron containing large interlocking, continuous graphite particles will lose strength and hardness more rapidly than harder irons containing discontinuous graphite particles, or carbide-forming alloys such as chromium, manganese (in excess of about 0.80 per cent), and molybdenum. In general, the lower the silicon and total carbon of the metal, the less will be the decrease in hardness and strength of the iron resulting from the above heat treatment and *vice versa*.

### *Heat Treatment of Car Wheels:*

Car wheels are given a heat treatment after casting for relief of strain. This subject is covered more fully in a subsequent section on White and Chilled Irons.

### *Softening for Machineability:*

It sometimes is necessary to soften castings containing some free cementite, particularly at edges and areas farthest from the gate area as evidenced by chilled areas with mottled or white fractures.

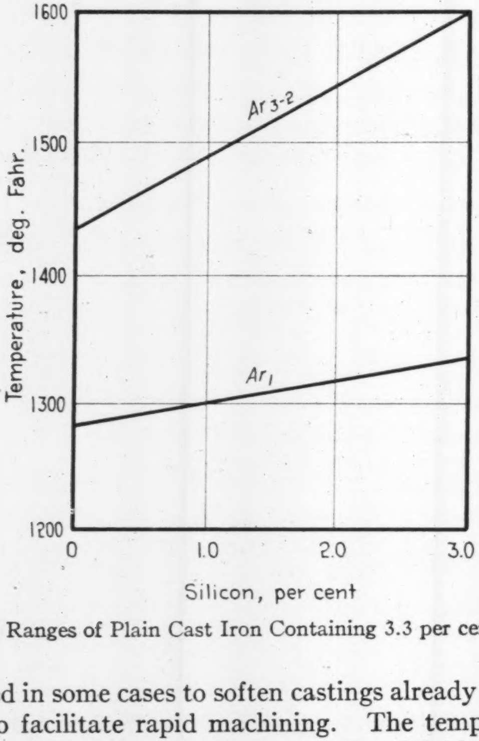


FIG. 63.—Critical Ranges of Plain Cast Iron Containing 3.3 per cent Total Carbon.

It also is desired in some cases to soften castings already gray throughout in order to facilitate rapid machining. The temperature range most commonly used is from 1400 to 1500 F. Highly alloyed irons are sometimes annealed at temperatures as high as 1800 F. In all annealing, care should be taken to prevent oxidation of the castings.

In general, softening for machineability is attended with a decrease in strength and combined carbon and an increase in graphite content. The length of time at temperature and the maximum temperature used are the determining factors for such changes for any given metal composition. The low-silicon, low-total-carbon and graphite irons resist the breakdown of structure more than high-silicon, high-total-carbon irons and require higher temperatures and

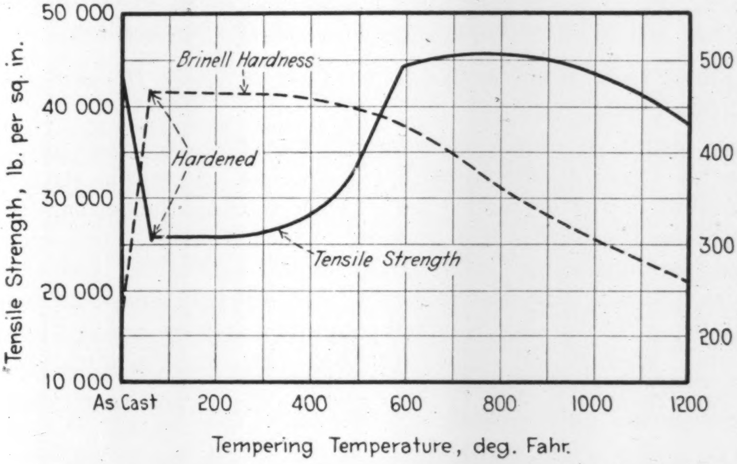


FIG. 64.—Effects of Tempering Temperatures on the Properties of Cast Iron.

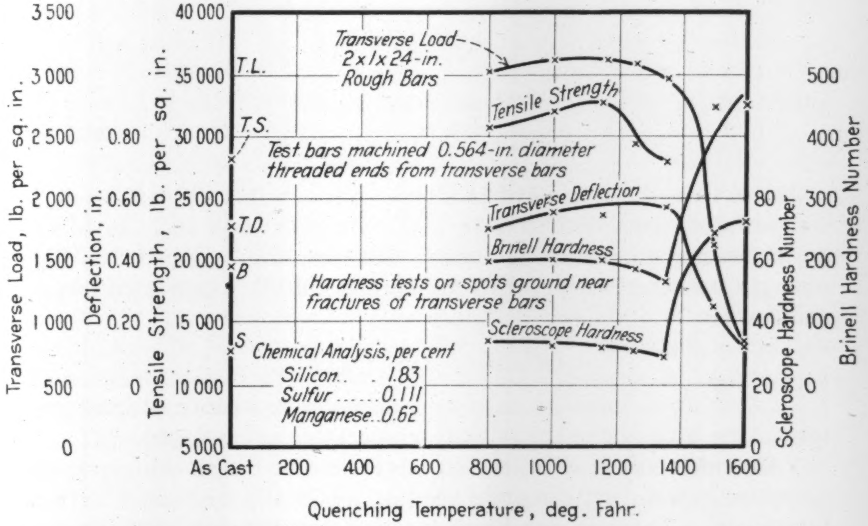


FIG. 65.—Effects of Quenching Temperatures on the Properties of Cast Iron. (Harper and MacPherran.)

Test specimens held at quenching temperature one hour before quenching in water.

longer time at temperature to get the same degree of softening. Irons containing carbide-forming alloying elements such as chromium are also much more resistant to annealing than the straight gray irons.

Completely annealed straight gray irons, that is, with no combined carbon, may have Brinell hardness values of from 120 to 130, whereas completely annealed alloy irons may have Brinell hardness values of 130 to 180 or higher, depending on the composition.

*Annealing Irons White or Mottled, as Cast, to Obtain Higher Strength Gray Irons:*

In order to obtain commercially machineable irons of high strengths (from 60,000 to 90,000 lb. per sq. in. tensile strength), irons white or mottled as cast are annealed to effect the decomposition of carbides into ferrite and graphite. Such irons are commercially called "semimalleable" and "short anneal malleable" but should not be confused with true malleable iron which is composed fundamentally of ferrite and rounded graphite particles called "temper carbon," whereas the irons described here are "semimalleable" irons which contain appreciable amounts of combined carbon and frequently are fully pearlitic. The total carbon usually is held at 2.00 to 2.80 per cent, with silicon content depending on section and not exceeding 1.80 per cent. Typical annealing cycles for this material are as follows:

Heat to 1700 to 1750 F.
Hold at temperature 1.5 to 3 hr.
Cool to 1375 F. in 3 hr.:
  (*a*) Oil quench and draw, or
  (*b*) Air cool, or
  (*c*) Cool slowly in furnace.

The Brinell hardness of this type of iron will vary, according to composition, from 150 to 200. Alloying elements such as nickel, molybdenum, etc., may be used. The resulting product has high strength, as noted above, with measurable ductility and good impact resistance.

## QUENCHING AND DRAWING

In order to increase wear and abrasion resistance of cast iron, quenching from above the critical temperature is employed.

Common soft gray irons that have a coarse interlocking graphite structure and a ferrite matrix are not materially improved by heat treatment. They do not harden satisfactorily, while drawing may or may not improve the strength properties. Small castings that have a fine graphitic structure and a matrix containing a large proportion of pearlite may be improved provided that heat treatment is carried out skillfully. There always remains the danger, however, that in the presence of high silicon, the heating process may decompose the silico-cementite into graphite and ferrite causing softening instead of hardening.

To heat treat satisfactorily, the graphite flakes should be small, well distributed and should not interlock, while the matrix should be wholly pearlitic. The graphitic carbon content of suitable heat treating cast irons should approximate 2.0 to 2.4 and the combined carbon 0.60 to 0.90 per cent.

Given these conditions, gray-iron castings respond to quenching and tempering much in the same way as carbon tool steel. The hardness increases in direct relation to the rapidity of quenching from above the critical temperature. Figure 63 shows the critical ranges of plain cast iron. Softening after hardening proceeds uniformly as the drawing temperature rises above 350 F. Drawing below 350 F. has little effect on the hardness. The strength properties—tensile, modulus of elasticity and impact resistance—are definitely improved. This is shown graphically in Fig. 64. The effect of quenching temperature is shown in Fig. 65.

Hardening is always accompanied by an appreciable loss of strength. A recovery of the original strength of the casting usually is obtained on drawing at above 500 F., and as the drawing temperature increases to 950 F. to 1000 F. the tensile and impact properties are improved from 20 to 50 per cent, depending upon the physical constitution and the structural disposition of the graphite in the original casting.

Heating of the castings for hardening should be carefully done. The castings should be heated slowly and uniformly to the quenching temperature. They should be held at the quenching temperature a sufficient length of time to insure that all parts are at the maximum or quenching temperature. A safe minimum is 30 minutes for comparatively small castings. The castings, if small and of symmetrical design, may be preheated to from 300 to 500 F. in one furnace and then transferred to another furnace which is held at the quenching temperature, with little danger of cracking, to promote economy of operation.

The quenching temperatures used on most irons in production at the present time are from 1450 to 1550 F. The quenching medium most commonly used is oil although water also is used to a limited extent. The quenching medium used is dependent on the composition of the metal. With some compositions the castings may be quenched in an air blast or still air and the desired hardness secured. Delbart(115) shows the advantages obtained in quenching of low-carbon irons with high manganese content. Manganese lowers the transformation point during the heating period, particularly when the manganese content reaches 2 per cent. This lowering of the critical range during the cooling period is still more marked when the manganese reaches 2 per cent, at which point the castings are susceptible to hardening by cooling in still air. Delbart obtained excellent results with irons having 2.5 to 2.8 per cent total carbon and 2 per cent manganese. Hurst also has called attention to the value of higher manganese in the heat treatment of cast iron. Other investigators have shown that with 1 per cent manganese and 1 to 2 per cent nickel, a marked effect on quenching is noticeable. That is because the influence of manganese and that of nickel are in the same direction and additive.

The composition of the material is largely governed by requirements of machineability in the as-cast condition. Castings to be hardened are usually rough machined before hardening and drawing and are finished by grinding. A small amount of growth occurs on quenching, and allowance must be made for this in leaving stock for finish grinding. Where strength properties and shock resistance are not of particular importance, the drawing operation may be omitted.

Some data on the wear resistance of hardened cylinder liners used in heavy duty internal combustion engines are available which indicate that hardening increases wear resistance from 2 to 7 times as compared to the wear resistance in the as-cast condition. Cylinder liners in this type of service have been in use both in the United States and England during the last two years only, so that final performance data are not yet available.

Brinell hardness values up to 600 may be obtained by hardening, depending on the composition of the metal employed. Extremely high hardness values are obtained on hardening irons not classed as machineable in the as-cast condition.

The alloying elements, nickel, molybdenum and manganese, lower the critical transformation points on cooling while chromium increases them. All four decrease the critical quenching speed and permit the formation of martensite with slower cooling. The final hardness values obtained are dependent on the average hardness of the matrix and the graphite particles present. It should be again noted that size, shape and distribution of graphite particles are extremely important.

The metal may be completely or partially annealed before hardening without appreciably affecting the response to quenching or hardening properties provided coarse, interlocking graphite is not present.

Care should be exercised in respect to sudden changes of temperature, both in heating and quenching, particularly where castings are irregular in section and where size is large.

### SPECIAL HEAT TREATMENT—NITRIDING

The nitriding process has been in some use(96, 97) for the past year or two to obtain surface hardness on finish machined castings of from 800 to 950 Brinell. Parts requiring extremely high wear resisting properties may be economically nitrided. The nitriding process consists of subjecting the castings, at a temperature of from 950 to 1000 F., to ammonia gas for a period of time, usually 60 to 90 hr.

Low total carbon, chromium, aluminum irons, were originally developed for nitriding purposes, somewhat similar to the steels used for nitriding treatment. It is claimed that chromium-vanadium irons also respond to nitriding treatment satisfactorily. Nickel also is used in nitriding irons to increase strength and toughness.

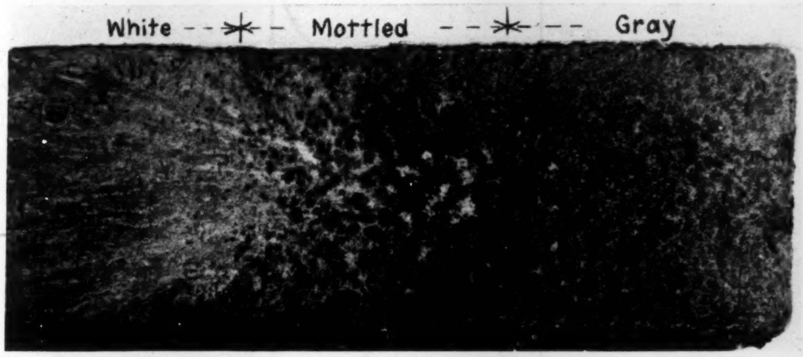


FIG. 66.—Section of Casting Showing White, Mottled and Gray Iron. (R. S. MacPherran.)

## VI. WHITE AND CHILLED IRONS

### GENERAL

A "white iron" is a cast iron in which practically all of the carbon is in the combined form. The purpose of chilling an iron casting is to produce on the chilled surface a layer of iron of a desired depth in which the carbon is all combined. This layer of course is harder than the remainder of the casting and will increase the resistance to abrasion or wear. The formation of white iron, chilled iron, or iron in which the carbon is all combined is made easier by the accelerated cooling due to the metal chills against which the melted iron is poured. The sudden cooling tends to keep the carbon in the combined form.

Among the many chilled and white iron castings in industrial use are those used in crusher heads, concaves, wearing plates, liners, drums, car wheels, plow shares, truck wheels, balls, stamp shoes, and dies, chilled rolls for rubber, grain, linoleum, paper and metals, draw dies, toggle plates, guides, cams, chute plates, and many other castings in which resistance to abrasion is a prime factor.

Chilled-iron castings usually show three distinct zones of cast structure: White, mottled and gray iron (Fig. 66). These three types of structure are controlled and influenced by the following factors: (*a*) Chemical composition, (*b*) Pouring temperature, (*c*) Thickness of casting in the chilled section, (*d*) Temperature of chill, and (*e*) Thickness of chill.

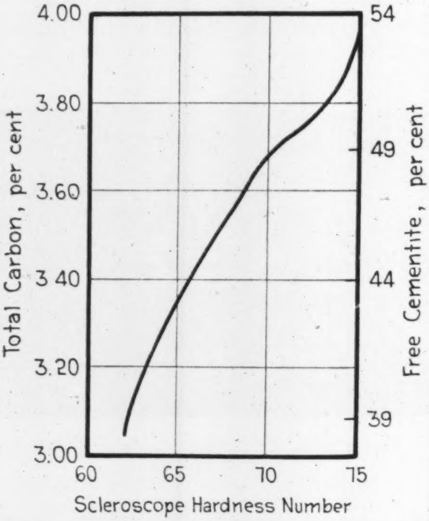


FIG. 67.—Relation of Total Carbon to Scleroscope Hardness. (J. Ehrler.)
Plotted from results on about 200 cupola and air furnace irons.

The desired depth will depend on the service to which the casting is to be subjected. The white or chilled portion of the casting increases resistance to wear but lowers the resistance to shock or impact. A casting with a too deep layer of chilled iron may therefore have a high resistance to wear but may give a poor service due to breaking under impact. For service requiring considerable resistance to impact, the casting is often made with one-third or more white iron on the chilled side and with a gray-iron back, of over one-third the section of casting. The center is of a more or less mottled iron and of a thickness depending partly on composition and partly on section of casting.

In making white-iron castings no chills are used and the cooling takes place more or less equally from all sides. The iron is so adjusted in composition that the outer layers are white, usually leaving a mottled or gray-iron center to strengthen the casting, and give the desired resistance to impact or shock. The proportion of gray iron so left will be determined by the kind of service required. In some special cases, castings are made white throughout.

The desired depth of chilled or white iron usually is obtained by varying the silicon and total carbon. If other conditions are the same, lowering the percentage of silicon or percentage of total carbon will tend to increase the depth of chill and raising the percentage of silicon or total carbon will tend to decrease such chill. A high-carbon iron usually is coarse-grained and a low-carbon iron, fine-grained.

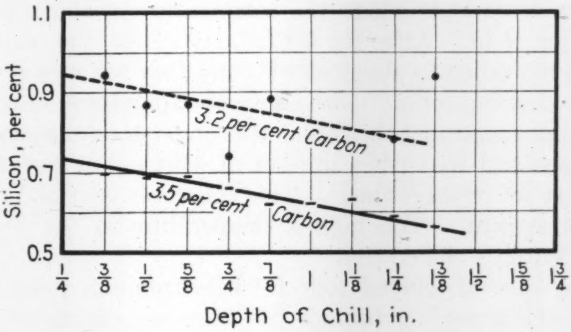


FIG. 68.—Influence of Carbon and Silicon on Depth of Chill. (J. Ehrler.)

Anything that will retard the cooling, such as increasing the size or thickness of casting, or withdrawing the casting from contact with the chill during critical period will also tend to lessen depth of chill.

As the silicon content is increased, the combined carbon is decreased due to the graphitizing action of the silicon. As the speed of cooling is increased, as by chilling, sufficient time may not be allowed to make this influence effective. In general, any iron is hard if cooled fast enough and any iron is soft if cooled slowly enough.

High manganese or sulfur increases the tendency to chill. Chromium has a decided chilling effect. Combinations of nickel and chromium are frequently used. The effect of total carbon on hardness is shown in Fig. 67. The influence of carbon and silicon on depth of chill is shown in Fig. 68.

## HEAT TREATMENT

Heat treatment by quenching and tempering is rarely applied to chilled or white iron castings. The chilled surface has already been subjected to an accelerated cooling. Too sudden a change in temperature also might result in cracking the casting.

Various types of chilled or white-iron castings, however, are given a heat treatment after casting for relief of strain. One of such treatments, sometimes given to car wheels, is as follows:

After the wheels have cooled in the molds to about 1400 F., they are removed from the mold and placed in a covered pit—this pit is kept at 700 F. to about 900 F. and may be preheated by oil burners if necessary. The wheels should not be placed in a cold pit. After holding in the pit for 24 hr., the temperature of the wheels has fallen to about 1000 F. They are then removed from the first pit and placed in a second pit where the temperature is about 600 F., and again left for 24 hr. After the second day's hold, the temperature of the wheels has fallen to about 650 F., and they are now removed and taken to a cleaning room. The above treatment will remove casting strains without softening the chill. It is understood that the operation is continuous and that when one set of wheels is removed from any pit, the next set replaces them. This conserves heat and facilitates a uniform treatment. Too high a temperature in the first pit will, of course, soften a chilled iron.

In the case of alloyed rolls (nickel-chromium rolls) a retarded cooling in the annealing furnace has been recommended when such rolls are inclined to crack or split. After the roll is cooled down to a temperature below the eutectoid point, it is stripped (removed from mold) and put in an oven where it is cooled at a rate slower than in the mold.

## HARDNESS

The scleroscope hardness of ordinary cupola white iron is from 60 to 65. The scleroscope hardness of ordinary alloy-free cupola chilled iron is from 65 to 75.

Where alloy elements are used, especially chromium and nickel, the scleroscope hardness is increased. In one alloy with nickel about 5 per cent and chromium $2\frac{1}{2}$ per cent, the scleroscope hardness of 95 to 100 with a Brinell hardness of 650 may be obtained. Great wear resistance is claimed for this type. The cost, however, is considerably increased. Hardness also may be obtained by the use of chromium; the nickel is added to increase toughness.

No definite relationship between hardness and length of wear has ever been established.

## EFFECT OF ALLOYS ON CHILLED AND WHITE-IRON CASTINGS

*Chromium.*—When used without other alloys, chromium increases the depth of chill and, by increasing the carbides, the hardness also is increased. Chromium frequently is alloyed with nickel, molybdenum or copper, and some investigators suggest a chromium-aluminum alloy.

*Nickel.*—Nickel is rarely used in chilled castings without another alloy—usually chromium. When used alone, it reduces the depth of chill. The core is strengthened due to a more even distribution of the graphite. Nickel reduces chill nearly one-third as much as an equal amount of silicon.

*Nickel and Chromium.*—Nickel and chromium should be considered alloyed together as well as separately. It is usually considered that one counteracts the bad effects of the other and supplements the good effects. A correctly alloyed nickel-chromium iron produces a hard close-grained casting that is very resistant to wear. Recently a nickel-chromium white iron of considerable commercial importance was introduced. This alloy is known under the trade name of "Ni-Hard" and properties comparing non-alloy and "Ni-Hard" irons are shown in Table XXXVII. Tests indicate that "Ni-Hard" will give better wear resistance and is tougher than the non-alloy chilled or white irons.

TABLE XXXVII.—TYPICAL RESULTS OF TESTS OF CHILLED PLAIN AND NI-HARD IRONS.[a]

| | PLAIN IRON | | NI-HARD | |
|---|---|---|---|---|
| | HIGH CARBON | LOW CARBON | HIGH CARBON | LOW CARBON |
| Brinell hardness number of chilled surface | 500 | 400 | 650 to 700 | 575 |
| Tensile strength in chilled section, lb. per sq. in. | 35 000 to 40 000[b] | 48 000 to 53 000[c] | 55 000 to 60 000[c] | 70 000 to 80 000[c] |
| Tensile strength in grey core, lb. per sq. in. | 16 000 to 25 000 | 22 000 to 39 000 | 30 000 to 35 000 | 40 000 to 50 000 |
| Total carbon, per cent | 3.50 | 2.75 | 3.50 | 2.75 |
| Silicon, per cent | 0.75 | 0.75 | 0.75 | 0.75 |
| Nickel, per cent | .... | .... | 4.50 | 4.50 |
| Chromium, per cent | .... | .... | 1.50 | 1.50 |

[a] Nickel Cast Iron Data, Section 3, No. 2, International Nickel Co., New York.

[b] Calculated from transverse tests.

[c] Test pieces ground from chilled blocks 0.800 in. in diameter A.S.T.M. Standard.

*Molybdenum.*—Molybdenum in chilled iron increases the strength but does not apparently affect the hardness. Molybdenum, however, does increase the wear resistance to some extent.

A copper-molybdenum alloy has proven satisfactory for rolls requiring strength and heat conductivity.

*Vanadium.*—Vanadium in white and chilled cast irons is of value in the refining of the dendritic structure, thus producing a tougher metal as indicated by impact tests.

*Copper.*—Copper tends to reduce depth of chill. It is seldom used alone as an alloying element. The usual combination is copper-molybdenum or chromium-copper-molybdenum.

*Aluminum.*—Aluminum as an alloying element in chilled iron has not been completely investigated. It is definitely known that it is a strong graphitizing and deoxidizing agent, so it therefore reduces the depth of chill.

Aluminum is only half as effective in reducing white iron to gray as silicon. One decided disadvantage, reported by Smith and Aufderhaar(104), to the use of aluminum in iron is the fact that considerable gas is evolved during the pouring operation, causing blowholes.

## VII. WELDING

Welding cast iron now is widely used for repair work and for various types of construction because modern methods give reliable results. Recent developments indicate that greatly enlarged applications of welding cast iron may be expected in the next few years, as methods and materials are still further improved.

The difficulty in welding cast iron is the hard area around a weld. This is caused by the solution of the graphite in the gray iron into the weld metal, and by the sudden cooling of the iron. Hard carbides may be formed when the iron cools, leaving unmachineable areas and sometimes cracks. Methods for combating this are mentioned below.

### GAS WELDING

The two general methods of welding cast iron by the oxyacetylene process are: Fusion welding with cast-iron rod; welding with bronze rod.

*Fusion Welding with Cast-Iron Rod:*

This method will give excellent machineability if the following precautions are taken. The whole casting should be preheated before welding, welded while hot, and allowed to cool very slowly. Because of the preheating, due allowance must be made for the effects of contraction and expansion.

*Preparation.*—To prepare the casting for fusion welding, the edges of the break should be beveled 45 deg. to within about $\frac{1}{8}$ in. of the bottom, thus making a 90-deg. V, as is shown in Fig. 69. The beveled edge should be cleaned by grinding or brushing to remove scale, rust, grease, or dirt. If necessary, the parts should be lined up.

*Welding.*—When the casting is heated to a dull red, it is ready for welding. The operator should begin by melting down the sides and bottom of the V to form a puddle about 1 in. long. The welding rod, heated nearly to the melting point, is inserted in this puddle, with care to keep the tip of the rod always below the surface of the weld metal. The proper position is shown in Fig. 70. Occasionally the rod should be removed from the puddle, dipped in the flux can, and

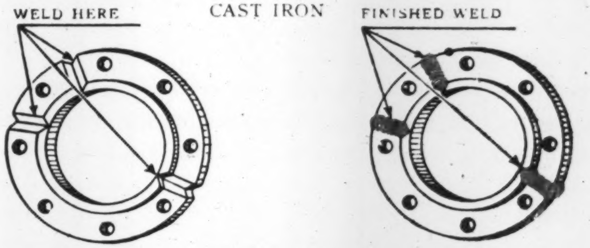


FIG. 69.—Proper Beveling for Welding Cast Iron. (Linde Air Products Co.)

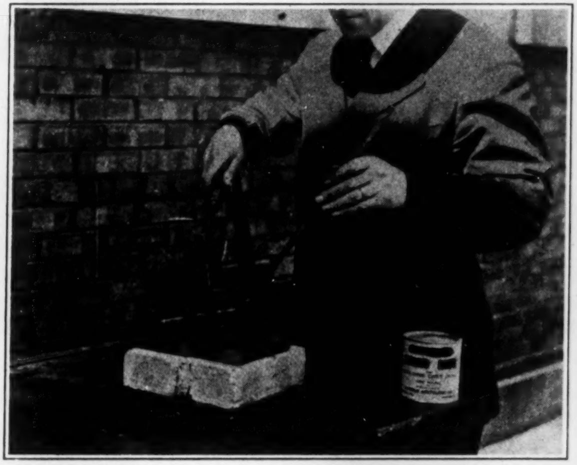


FIG. 70.—Proper Position for Welding Cast Iron. (Linde Air Products Co.)

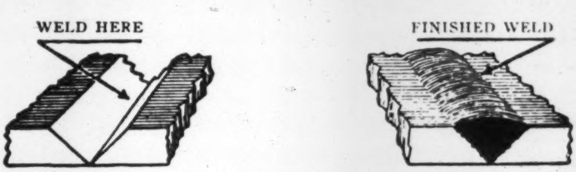


FIG. 71.—Proper Filling-In the Weld. (Linde Air Products Co.)

returned. Gas bubbles and bright spots are indications of impurities and should be worked to the surface by vigorous manipulation of rod and blowpipe assisted by as much flux as necessary. When this 1-in. section has been built up slightly above the adjacent surface (as shown in Fig. 71) the operation is repeated on the next section, and the weld completed as quickly as possible.

Since quick cooling will produce a white-iron structure, castings should be cooled very slowly. Large castings may be cooled in the preheating furnace; small ones are best cooled in some heat-insulating material such as dry asbestos fiber, cement, or slaked lime. Figure 72 shows the structure of a properly welded cast iron, cooled slowly.

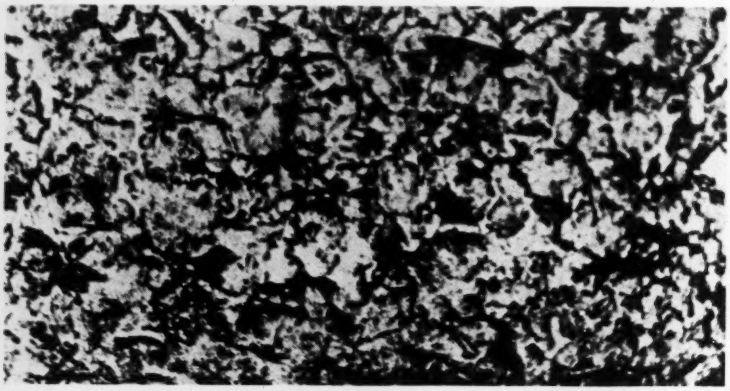


FIG. 72.—Structure of Weld Metal. (Linde Air Products Co.)

*Bronze Welding:*

Bronze welding does not affect the properties of the casting because it is done at a temperature below the fusion point of iron. With this method it is not necessary to preheat the entire casting, although parts sometimes are preheated before welding. Bronze welding is used extensively for the repair of cast-iron parts and for building up the surfaces of castings.

*Preparation.*—Beveling is not necessary but is advisable for butt joints over ⅛ in. thick. The edges to be joined or built up should be cleaned by brushing or grinding and by washing with a grease solvent such as gasoline.

*Welding.*—A slightly oxidizing flame and the proper flux are most important. To commence welding, a small area should be heated just hot enough so that metal from the fluxed bronze rod will spread out in a thin layer over the base metal to produce the "tinning" coating, which provides the strong bond between the base metal and the bronze. On heavy jobs of welding, it is frequently necessary to deposit the bronze in layers. If good tinning is obtained in the first layer, a strong weld will result through thorough fusion of the welding puddle and previous layers of bronze. The strength of properly made gas welds is equal to the strength of the original metal.

Due to the increased reliability of methods, the use of gas welding on cast iron continues to become more widely practiced all the time, particularly for repair work. In the foundry, castings are frequently produced which have small surface defects, in no way interfering with their serviceability, but detracting from appearance. Such defects can be readily and safely remedied by means of gas welding. Worn machinery is often made again serviceable by building up the worn parts to original dimensions. Automotive repair work, such as cracked cylinders, etc., makes use of the gas-welding process.

## ELECTRIC WELDING

Cast iron can be welded successfully by the electric-arc process, but much depends on the nature of the casting, the type of weld made, and the technique of the operator. The chief difficulty in arc welding is breakage due to weak welds. This weakness is caused by (1) poor fusion; (2) formation of white iron at the fusion zone; and (3) production of large stresses at the weld.

Since cast iron is a relatively poor conductor of heat and offers considerable resistance as an electrical conductor, it is hard to use as an electrode. Therefore, steel or non-ferrous electrodes are in general use. As is well known, a hard area of white iron is formed when gray iron is heated to the fusing temperature and suddenly cooled. This effect is accentuated by the action of the molten-steel electrode, which dissolves the graphite out of the gray iron immediately adjacent to the weld, so that there is present not only hard, brittle white iron, but also hard, brittle high-carbon steel.

When steel cools, it contracts a great deal more than cast iron, the center of a steel weld having 50 per cent more contraction than the cast iron around it. Naturally, this difference in contraction sets up stresses at the weld which may result in cracks and breaks.

### *Electrodes:*

For strength and cheapness, mild steel electrodes are used. Where machineability is desired, non-ferrous electrodes such as Monel metal and high-nickel electrodes are best. Monel and nickel wire are frequently employed for filling in sand holes. If the weld is deep, steel rod may be used where strength is important, and the nickel-alloy rod at points which must be machined.

*Preparation:*

*Studding.*—A popular method of reinforcing welds in cast iron is "studding," or placing steel studs in small threaded holes. This is illustrated in Fig. 73, showing a large cast-iron shear, studded ready for welding. The deposited weld metal fuses to the studs, and after the weld is made, the studs stretch slightly, thus relieving the strains in the iron and preventing cracks.

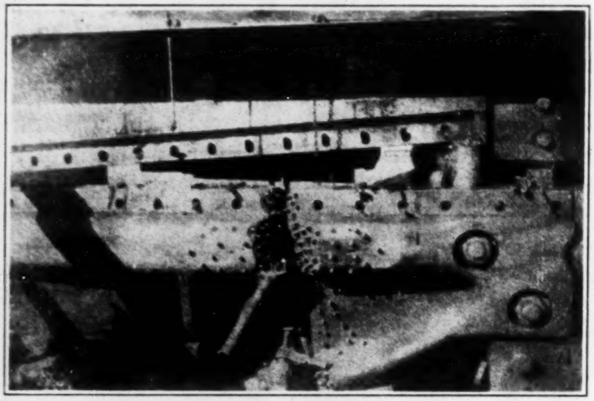


FIG. 73.—Steel Studs in Cast Iron Shear Prepared for Welding. (General Electric Co.)

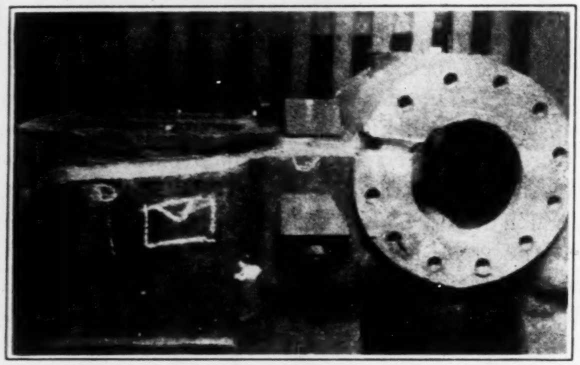


FIG. 74.—Crack Chipped Out and Grooved for Welding. (General Electric Co.)

*Preheating.*—Preheating the castings to a dull red heat lessens the difficulties of welding, promotes better fusion, relieves strains, and reduces the tendency towards cracking and unmachineable spots. An anneal after welding serves the same purpose, especially as to softening hard spots. Operation at very low temperature, which means using an electrode with low melting point, and welding very slowly is preferred to preheating by many, and gives good results.

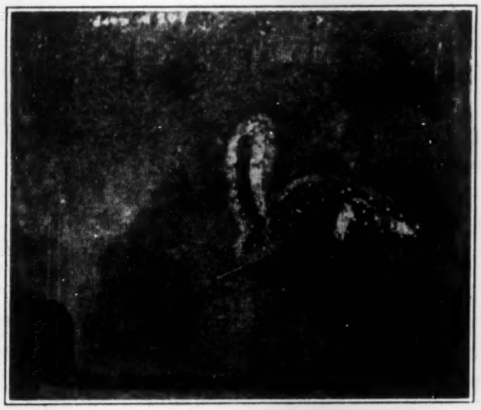


FIG. 75.—Casting Ready for Welding. (Lincoln Electric Co.)

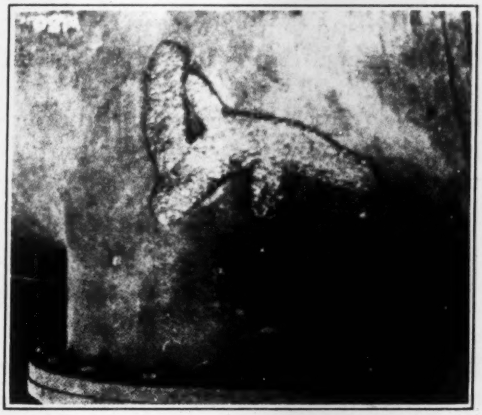


FIG. 76.—Casting After Welding. (Lincoln Electric Co.)

*Cleaning.*—All surfaces should be extremely clean. The sand and scale is chipped off, and a groove usually is formed. Figure 74 shows a broken feed-water pump with the crack chipped out and grooved ready for welding.

*Welding:*

If preheating has not been used, welding should proceed slowly, and the castings should be kept as cold as possible. It is best to deposit a single layer over the entire cast-iron surface to be covered, and then to fill flush with the rest of the casting. A very short bead should be deposited, only an inch or so at a time, and the metal allowed to cool, in order to reduce to a minimum the transverse stress between weld metal and cast iron. Each bead should be peened white hot, using either a machinist's ball-and-peen or an air hammer, to spread the weld metal and relieve strains. After one bead has been deposited and peened, the next should be put down some space away to avoid overheating the casting. In practice, an operator should be able at all times to keep his fingers on the metal 2 in. away from the weld.

Another method recently developed uses coated cast-iron electrodes which have been so processed that they will flow under the arc with reversed polarity. No attempt is made to keep the casting cool; in fact, a high current with considerable heat is used. For thin sections, a copper-alloy rod is recommended, instead of Monel metal or nickel.

Properly welded castings will have a strength approximately equal to that of the original cast iron. When carefully made, they should have no cracks or hard spots.

An example of electric welding for repair work is shown in Figs. 75 and 76. Figure 75 shows the casting with the crack cleaned, chipped out, and studded, preparatory to welding; Fig. 76 shows the completed weld.

## VIII. FOUNDRY FACTORS OF IMPORTANCE IN THE PRODUCTION OF IRON CASTINGS

The design of a part covers control of (1) outline or shape, (2) thickness of prevailing section, and (3) variations in sectional thicknesses. There are, however, some other very important considerations confronting the designer, such as choosing the material which will be most satisfactory and the method of production, which have a great influence on the design. Elsewhere in this symposium are presented data on the properties of various types of cast irons from which the engineer can select that most satisfactory for his needs.

Consideration of production methods has been especially stressed in designing parts to be made by drop forging, stamping, welding and extrusion. However, comparatively little attention has been given to designing for castings, largely because of a traditional attitude that castings can be made in any form. While this may be true in a certain sense, yet to produce castings in the most economical manner and with the best properties, there are certain foundry factors which the designer should keep in mind. This has been well illustrated in recent cases where weldings have been used to replace castings, and designs have been simplified to meet the limitations of welding. In many of these cases, the foundryman has shown that, given the same latitude and cooperation in redesigning, he can produce the same part as economically or more so and at the same time produce the part of better quality as a casting. This question of designing for castings has been covered in several recent articles which are referred to in the bibliography, references (105) to (114), inclusive.

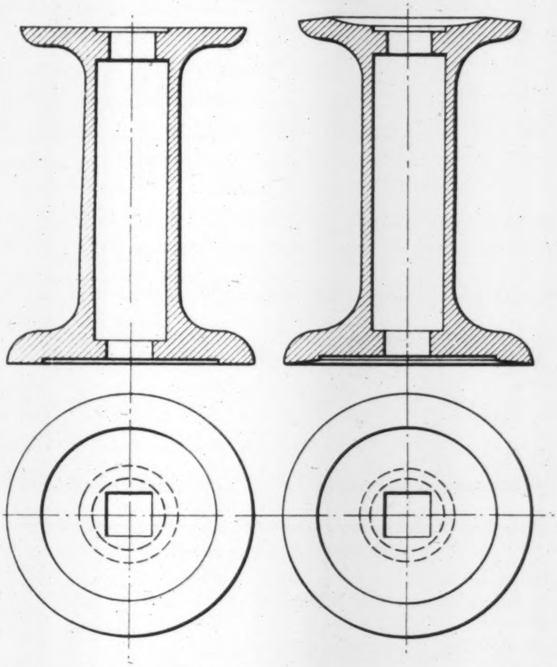


FIG. 77.—Example of Spool Casting. (H. Bornstein.)

In its original form (left) the casting had excessive breakage in the flange. Redesigned (right) weight was reduced but castings resisted twice as much service load as the original. The flange was made heavier but the body section was reduced to make a lighter total weight casting.

The elimination of excessive weight is a particularly important problem that frequently confronts the engineer today. There are innumerable instances where lighter weight in manufactured products has resulted in great improvement in performance and efficiency (Fig. 77) and increased salability. In the past few years there have been developed cast irons of greatly improved strength properties which, if used by the engineer, will enable him to save weight in his designs. Data on these improved irons are given elsewhere in this symposium.

To emphasize some of the more important specific foundry factors affecting design, the following recommendations are given to assist the engineer in designing castings to the best advantage.

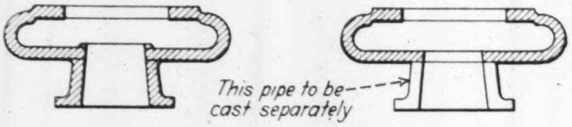


FIG. 78.—Example of Casting when, by Cooperation Between Designer and Foundry, Castings Were Made More Efficiently Without Affecting Utility. (W. W. Kerlin.)

The original one-piece casting (left) required a large flask and special molding features. Redesigned, the part was made as two castings, simplifying molding and reducing cost of production.

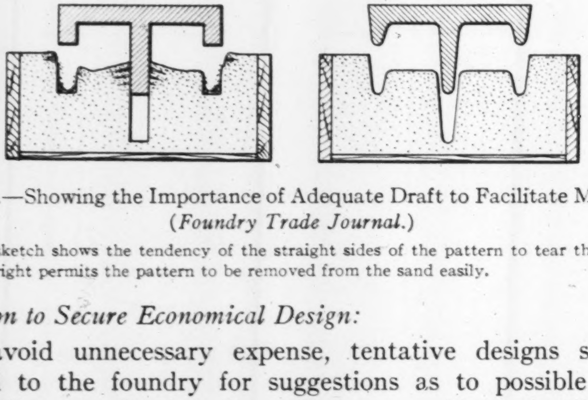


FIG. 79.—Showing the Importance of Adequate Draft to Facilitate Molding. (*Foundry Trade Journal.*)

The left sketch shows the tendency of the straight sides of the pattern to tear the mold. The design at the right permits the pattern to be removed from the sand easily.

*Cooperation to Secure Economical Design:*

To avoid unnecessary expense, tentative designs should be submitted to the foundry for suggestions as to possible changes, which, while not affecting the utility, may aid in simplifying the foundry practice and make a better or a cheaper casting, or both (Fig. 78). Either the designer or the foundryman must visualize the molding methods applicable to the design and make such modifications as may be necessary to insure a good product.

The simplest form of casting, the ingot, has been given every attention by the producers of ingots for the promotion of soundness, with results that have justified the efforts. All other castings, which are in fact modified forms of ingots, present more complicated metallurgical problems in prevention of shrinkage cracks, blows and washes than do ingots.

*Draft:*

Draft is the taper that must be allowed on all vertical faces to permit removing the pattern from the sand without excessive rapping and tearing of the walls of the mold which requires subsequent patching (Fig. 79). Regardless of the type of pattern equipment, draft must be considered in all pattern designs. Drawings should specify whether this draft is to be added to or subtracted from casting dimensions as given, because such draft frequently affects the subsequent use of the castings.

The amount of draft needed will depend on the shape and size of the pattern and the method of production, whether by hand or machine. Machine molding will require the minimum amount. Interior portion usually will require more draft than exterior surfaces.

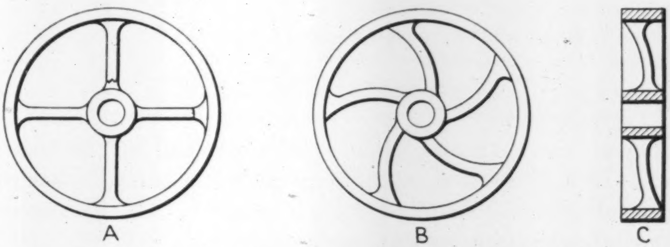


FIG. 80.—Example of Redesign to Allow for Adjustment Due to Contraction. (W. W. Kerlin.)

Showing contraction adjustment provided by the curved spokes (*B*) and by use of 5 spokes instead of 4 as shown in a common faulty design at (*A*). An alternative method to (*B*) to secure the same results is shown in (*C*).

Under normal conditions *the amount of draft recommended is not less than $\frac{1}{16}$ in. per ft.*, this allowance to be made in the design dimension. Metal thickness, however, shall be maintained if possible.

*Shrinkage, Patternmaker's:*

Shrinkage allowance for patterns is the correction that must be made for change of volume as the solidified casting cools in the mold, the contraction taking place as the casting cools from the *freezing* temperature of the metal to *room* temperature. This shrinkage is compensated for by making the pattern larger by the amount the particular metal used shrinks in cooling. The shrinkage allowance commonly is known as *patternmaker's shrinkage.* This shrinkage varies according to the design of the casting and the temperature and composition of the metal poured, as well as the restraint of the mold caused by projecting lugs, cores, etc. Where a casting is restricted from contracting normally as by flanges at an angle to the contracting direction, sometimes it is necessary to provide against the cracking of the casting. Various methods are used. Figure 80 illustrates one where the design was changed.

Many metals have several different allowances for the different dimensions owing to this variation in the shrinkage of the metal, especially if of high-test cast iron when cooling in the mold. A normal shrinkage for gray cast irons with tensile strengths up to 38,000 lb. per sq. in. is $\frac{1}{8}$ in. per ft. A recommended shrinkage scale, taking into account the length and construction of patterns, has been formulated for gray cast irons as given below:

| | |
|---|---|
| Patterns up to 23 in., open construction | $\frac{1}{8}$ in. per ft. |
| Patterns from 25 to 48 in., open construction | $\frac{1}{10}$ in. per ft. |
| Patterns above 48 in., open construction | $\frac{1}{12}$ in. per ft. |
| Patterns up to 20 in., cored construction | $\frac{1}{8}$ in. per ft. |
| Patterns from 25 to 36 in., cored construction | $\frac{1}{10}$ in. per ft. |
| Patterns above 36 in., cored construction | $\frac{1}{12}$ in. per ft. |

*Machine-Finish Allowance:*

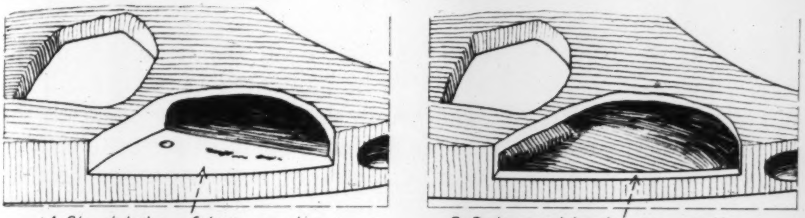
Sufficient excess metal should be provided for all machined surfaces. This allowance, commonly called "finish," depends on (*a*) the metal used, (*b*) shape of part, (*c*) size of part, (*d*) tendency to warp, and (*e*) machining method and set-up.

Machined surfaces, wherever possible, should be cast in the drag since then there is less chance of these surfaces containing defects such as shrinkage, dross and blowholes, than with those surfaces in the cope. Where such finished surfaces must be cast in the cope, an extra finish allowance should be made. There are some types of castings which can be cast to size if sufficient attention is given to molding sand and facing. A schedule of machining allowance is given below:

| | |
|---|---|
| Patterns up to 12 in | $\frac{3}{32}$ in. for bore $\frac{1}{8}$ in. |
| Patterns 13 to 24 in | $\frac{1}{8}$ in. for bore $\frac{3}{16}$ in. |
| Patterns 25 to 42 in | $\frac{3}{16}$ in. for bore $\frac{1}{4}$ in. |
| Patterns 43 to 60 in | $\frac{1}{4}$ in. for bore $\frac{5}{16}$ in. |
| Patterns 61 to 80 in | $\frac{5}{16}$ in. for bore $\frac{3}{8}$ in. |
| Patterns 81 to 120 in | $\frac{3}{8}$ in. for bore $\frac{7}{16}$ in. |
| Patterns above 120 in | Special instructions. |

*Locating Points:*

Locating points to be used by the machine shop should be indicated on the drawing so that castings may be satisfactorily checked, always from the same point of origin, by the pattern shop, foundry



A-Shrink holes of heavy section

B-Redesigned to give even casting

FIG. 81.—Redesign of Casting to Give Even Metal Sections. (H. Bornstein.)

The original design (left) had a very heavy section joining a light section causing shrink holes to appear in the heavy section.

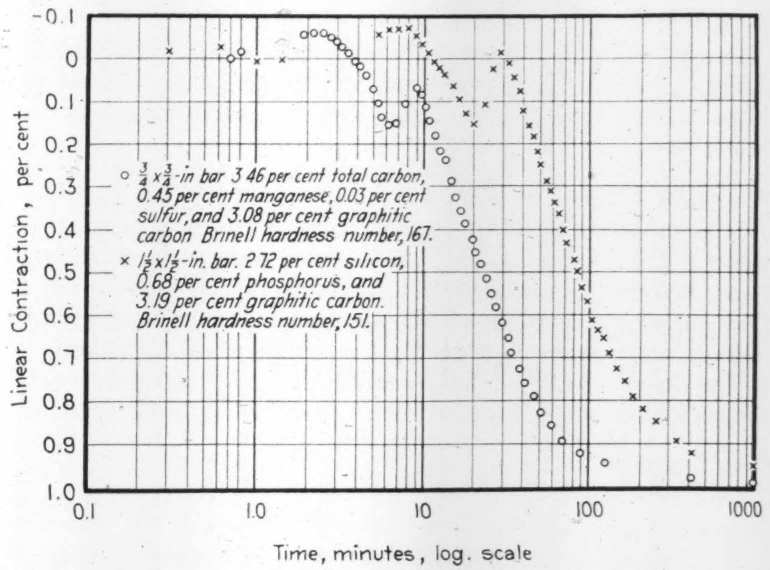


FIG. 82.—Curves Showing Relative Changes of Length in a ¾-in. Square Bar and a 1½-in. Square Bar, Each Contracting Independently. (Curves supplied by Foundry Section, U. S. Bureau of Standards.)

These curves emphasize the desirability of designing castings with as uniform section thicknesses as possible.

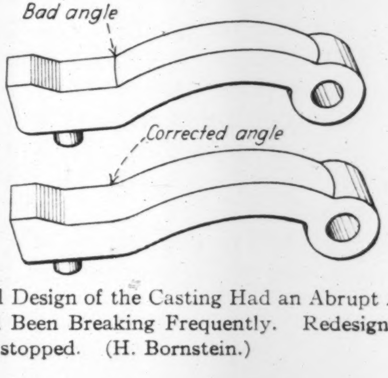


FIG. 83.—The Original Design of the Casting Had an Abrupt Angle at Which Point the Castings Had Been Breaking Frequently. Redesigned with a wide fillet, the breakage was stopped. (H. Bornstein.)

or machine shop. They should be placed on the same side of the parting line and so placed as not to be influenced by a core shift or shift of the drag or cope. The points should be as far apart as the size of the casting permits in order to insure the most accurate results. Dimensions which have no finish allowance and are to be held to very close limits, should be considered as the proper place from which to start development of tooling fixtures. The jig spots are very important items frequently neglected until the casting is made, with much subsequent loss.

### *Parting Lines:*

Parting lines should be made as even as possible to facilitate molding. Avoid irregular and multiple parting lines because they tend to molding complexities, dimensional irregularities and increased cleaning costs.

### *Provision for Proper Feeding:*

It is important at all times to provide for free flow of metal from one part of the mold to another. If there are some parts of the casting that are heavier than others, these heavy or thicker sections must be fed by sufficient metal through heads and risers, to compensate for shrinkage which takes place during the process of solidification.

The design should be made sufficiently heavy or thick to permit adding a gate of such size as to insure good feeding of all sections.

### *Section Uniformity:*

Uniformity of section thickness is very desirable from a foundry standpoint. This equalizes the rate of solidification, which is a very essential consideration for the manufacture of quality castings, and simplifies both the gating and the feeding. Non-uniform solidification sets up internal strains and causes shrinkage defects and cracks (Fig. 81). The reasons for the severe strains caused by differences in section are well illustrated in Fig. 82, which shows the relative changes of length in a $\frac{3}{4}$-in. square bar and a $1\frac{1}{2}$-in. square bar, each contracting independently, the contraction being plotted against the time. Where uniform sections are not possible, any changes in section should be made with a gradual taper, rather than abruptly.

Uneven rates of shrinkage between heavy and light sections may cause the metal to crack, result in shrinkage cavities, or give rise to directional or dendritic crystallization in the metal that does not lend itself to high strength.

A well-designed gray-iron casting may be said to be one that can be made commercially and whose sections are no thicker than is essential to the desired unit of strength and which are evenly proportioned to avoid local slow cooling. The cheapest design is that which presents least difficulties and risks in manufacture.

*Fillets:*

Size of fillets depends on (*a*) the metal used, (*b*) shape and thickness of wall section, and (*c*) the size of the casting. Adequate fillets at all intersections will materially reduce foundry scrap (Fig. 83) due to shrinkage and cracks at these points. Sharp angles are apt to produce sand washes in a mold. Fillets should not be so large as to interfere unduly with even metal section.

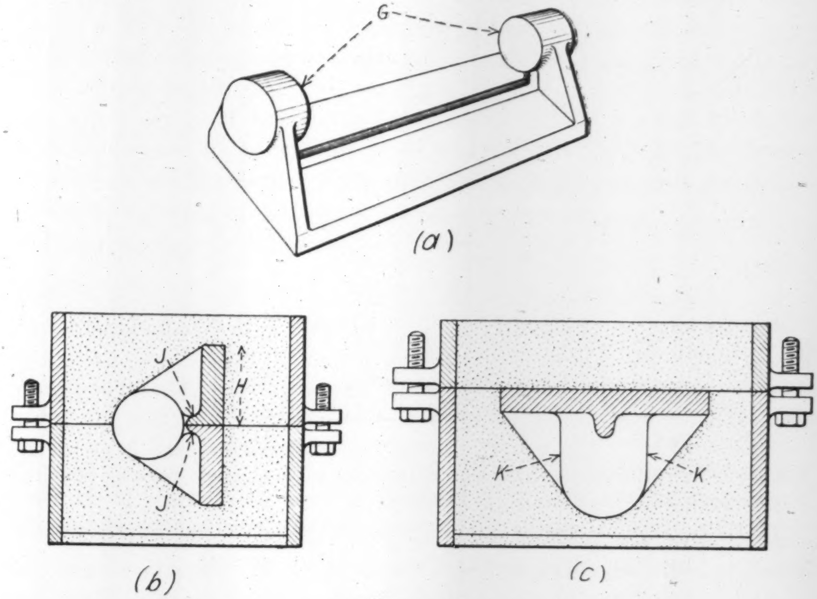


FIG. 84.—Redesign of Bosses to Improve Foundry Production. (*Foundry Trade Journal.*)

(*a*) Common design of bosses on a bracket at *G*. The design at *K* shows an improvement for foundry production as it facilitates molding and permits better feeding of the heavier section. (*b*) Molding method required for original design. (*c*) Molding method of redesign permitting all the casting to be set in the drag.

*Square Corners:*

The outer circumference of thin flanges should not be made with square corners if this can be avoided. This is especially true for white-iron castings or those gray-iron castings that are liable to produce chilled iron in thin sections. A sharp corner may readily develop a minute check that will extend into a crack, particularly in the vicinity of the gates where the metal cools last. In cases of this kind, losses due to cracks will be greatly lessened if the corners are slightly rounded off.

*Feeding Through Uneven Sections:*

A hub-like pattern may be very intricate, and of such a character that commercially and practically it must be gated on a flange that may be too thin as designed to permit of the use of gates thick enough to allow for proper feeding, even when feeding heads are used at available locations. A slight increase in thickness of flange would admit of the use of the proper size gates and the production of a sound casting. At the least, a local thickening of the web for the width of the gate should be permitted, provided this would not interfere with the assembling of the casting. In some cases the overall cost of the casting will be lowered by increasing the metal section because of the greater soundness obtained.

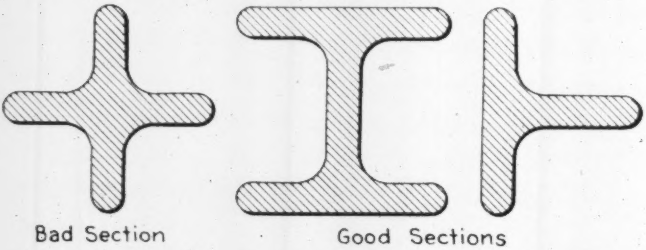


FIG. 85.—Rib Sections Such as Those Forming a Cross (Left Sketch) Should be Avoided Because There is a Tendency to Form Shrink Holes in the Heavy Section. Sections such as those shown at the right form stronger castings.

*Bosses and Lugs:*

Bosses and lugs are common in all castings and frequently lead to foundry difficulties. It is advisable to use a minimum of bosses and when used they should conform to the thickness of the adjacent section as closely as possible. Where they join onto a thinner section of a casting, non-uniform solidification must be provided against. This may be accomplished by a riser acting as a feed for the boss, or a metal chill may be placed against the face of the boss. Such a chill simply tends to equalize the rate of solidification and make it more or less identical with the thin section of the casting.

Bosses and lugs often are located on an interior portion of a casting, in which case the casting should be so designed as to provide heavy sections or ribs of metal leading to a feeding source. (Fig. 84.)

*Ribs:*

Ribs are used primarily as stiffeners and reinforcing members in general. The rib sections should have a fair relation to the casting section, inasmuch as the pouring temperature is governed to a considerable degree by the thinnest section in the casting. Ribs, also, because of rapid cooling, may set up casting strains and cracks and should be avoided wherever possible.

Where advisable, large flat areas may be ribbed to prevent distortion when cooling from high temperatures, although this depends upon the general design of the casting. Sections such as ribs forming a cross should at all times be avoided. (Fig. 85.)

*Pockets:*

Deep pockets and recesses in the casting should be avoided as much as possible and to minimize the use of cores and to make the molding operation less complicated and difficult.

*Holes:*

Holes on which no machine finish is required usually are cored, provided the tolerance is at least $\frac{1}{16}$ in. on the diameter. Large holes requiring machined surfaces frequently are cored and subsequently bored to dimension. It is to be noted, however, that it is often desirable and sometimes even necessary to avoid shrinks in bosses, to core out small holes which are later drilled.

*Proper Provision for Cores:*

Where the design calls for the use of internal cores, a means should be provided for egress of gases to the outside of mold during casting to obviate "blown" castings. Sufficient support to hold internal cores in their proper position also is very necessary. When cores are not properly supported through core prints, metal chaplets are used to support the core. The use of metal chaplets should be avoided when possible.

*Dry-Sand Cores:*

It is desirable, wherever possible, to avoid dry-sand cores, primarily because of the higher cost. Where the cooling metal contracts around a dry-sand core, strains and cracks may develop because the core does not yield sufficiently to the cooling metal as it contracts. Cores should be designed of sufficient thickness to be satisfactorily handled and set without excessive breakage. Sufficient thickness also is desirable to prevent the molten metal from burning through the core and increasing the cleaning cost.

### *Green-Sand Cores:*

Green-sand cores are preferable to dry-sand cores wherever it is possible to use them, because of lower cost, cheaper cleaning expense, etc. It is preferable, therefore, to have the design such that green-sand cores can be used.

### *Finishing:*

The design of a casting often is a serious consideration in the cleaning and trimming cost, which may far outweigh the molding cost. It is well for the designer to keep in mind the expense involved in cutting off risers in inaccessible spots.

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# SHEARING PROPERTIES AND POISSON'S RATIO OF STRUCTURAL AND ALLOY STEELS

By Inge Lyse[1] and H. J. Godfrey[2]

## Synopsis

The paper describes the results of tests of different grades of structural and alloy steels to determine shearing properties (modulus of elasticity, yield point and ultimate strength) and their relation to the corresponding tensile properties. Poisson's ratio was also determined. Shearing properties were determined from slotted plate specimens tested in tension and from solid and hollow cylindrical torsion specimens.

The results showed considerable variation in the ratio of yield point in shear to yield point in tension for the different steels. Shearing modulus of elasticity determined by direct measurement agreed reasonably well with values calculated from values of tension modulus and Poisson's ratio. The values of Poisson's ratio did not vary greatly for the different steels.

## Introduction

During a recent investigation of web buckling in steel beams, carried out at the Fritz Engineering Laboratory, Lehigh University, it was found that the ratio between the yield points in shear and in tension varied considerably for different grades of structural steel. In order to study the shearing properties more fully, the investigation reported in this paper was carried out, in which yield point, ultimate strength and modulus of elasticity in shear and in tension were determined and compared for a number of different grades of structural steel and ten heat-treated alloy steels. Poisson's ratio for these steels was also determined by direct measurement of lateral deformation of specimens under tension.

Since the shearing properties of steel are more difficult to determine than its tensile properties, several different methods of test were used as described hereafter in determining yield point and ultimate strength in shear. Shearing modulus of elasticity, $G$, was determined by the usual torsion test on solid cylindrical specimens, and by computation from the tension modulus of elasticity, $E$, and Poisson's ratio, $\mu$, using the following equation:

$$G = \frac{E}{2\,(1 + \mu)} \quad \text{.......................................} \quad (1)$$

which is theoretically true for homogeneous, isotropic materials.

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## MATERIALS

The several grades of structural steel comprised rolled I-beams, soft steel plates, tank steel plates, and $1\frac{1}{2}$-in. rolled rod. There were also included some soft, medium and hard steel rods of "structural" grade and a structural nickel steel.

TABLE I.—TENSILE AND SHEARING PROPERTIES AND POISSON'S RATIO FOR STRUCTURAL STEELS.

Values reported for moduli of elasticity in tension and shear and for Poisson's ratio are averages of two determinations. Values reported for strengths in shear and in tension are in general averages of four or more specimens; occasionally only two specimens of a kind were tested.

| Type of Steel | Tension | | | Poisson's Ratio, Observed | Shear | | | | Ratio, Yield Point in Shear to Yield Point in Tension |
|---|---|---|---|---|---|---|---|---|---|
| | Modulus of Elasticity, lb. per sq. in. | Yield Point, lb. per sq. in. | Ultimate Strength, lb. per sq. in. | | Modulus of Elasticity, lb. per sq. in.[c] | | Yield Point, lb. per sq. in. | Ultimate Strength, lb. per sq. in. | |
| | | | | | Observed | Calculated | | | |
| Webs of I-beams: | | | | | | | | | |
| No. 1 | ........ | 37 340 | 58 810 | ..... | ........ | ........ | 23 650 | ..... | 0.634 |
| No. 2 | ........ | 37 970 | 59 260 | ..... | ........ | ........ | 22 500 | 50 750 | 0.593 |
| No. 3 | ........ | 40 800 | 59 370 | ..... | ........ | ........ | 22 300 | 50 200 | 0.546 |
| No. 4 | ........ | 39 550 | 57 800 | ..... | ........ | ........ | 24 500 | 50 600 | 0.620 |
| WB-4 | ........ | 43 500 | 61 600 | ..... | ........ | ........ | 22 000 | 53 400 | 0.505 |
| WB-5 | ........ | 51 300 | 66 650 | ..... | ........ | ........ | 29 200 | 55 630 | 0.570 |
| CT (1-4) | ........ | 47 000 | 63 900 | ..... | ........ | ........ | 25 000 | 53 300 | 0.531 |
| CT (5-6) | ........ | 44 660 | 62 100 | ..... | ........ | ........ | 25 800 | 53 100 | 0.577 |
| Tank steel plates: | | | | | | | | | |
| WB-1 | ........ | 43 250 | 62 000 | ..... | ........ | ........ | 22 000 | 53 500 | 0.509 |
| WB-2 | ........ | 47 810 | 60 900 | ..... | ........ | ........ | 24 500 | 51 300 | 0.513 |
| WB-3 | ........ | 49 600 | 61 425 | ..... | ........ | ........ | 26 470 | 51 600 | 0.534 |
| Soft steel plates: | | | | | | | | | |
| WB-6 | ........ | 33 080 | 46 800 | ..... | ........ | ........ | 17 450 | 46 900 | 0.527 |
| WB-7 | ........ | 33 700 | 48 400 | ..... | ........ | ........ | 18 600 | 45 850 | 0.551 |
| WB-8 | ........ | 29 680 | 46 080 | ..... | ........ | ........ | 19 920 | 46 250 | 0.671 |
| WB-9 | ........ | 30 280 | 46 980 | ..... | ........ | ........ | 18 480 | 44 800 | 0.610 |
| WB-10 | ........ | 30 270 | 47 150 | ..... | ........ | ........ | 18 400 | 46 250 | 0.607 |
| S-1 | 29 200 000 | 30 650 | 60 200 | 0.292 | 11 330 000 | 11 300 000 | 19 590[b] | 54 400 | 0.638 |
| S-2 | 29 050 000 | 26 900 | 60 400 | 0.278 | 11 900 000 | 11 400 000 | 16 500 | 49 200 | 0.613 |
| Steel rods: | | | | | | | | | |
| 0.5-in. diam. | 29 000 000 | 35 350 | 60 150 | 0.302 | 12 600 000 | 11 100 000 | 22 500[b] | 68 800[b] | 0.637 |
| | 29 400 000 | ...... | ...... | 0.271 | [d] | 11 560 000 | | | |
| 1.0-in. diam. | 29 500 000 | 36 400 | 61 250 | 0.299 | 11 800 000 | 11 350 000 | 22 650[b] | 66 250[b] | 0.623 |
| 1.5-in. diam. | 29 600 000 | 39 350 | 62 500 | 0.302 | 12 400 000 | 11 350 000 | 24 700[b] | ..... | 0.628 |
| | 29 800 000 | | | 0.278 | [d] | 11 680 000 | | | |
| Soft steel | ........ | 30 000 | 55 000 | ..... | ........ | ........ | 20 200[b] | 61 400[b] | 0.674 |
| Medium steel[a] | ........ | 30 800 | 57 650 | ..... | ........ | ........ | 22 750[b] | 68 000[b] | 0.739 |
| Hard steel[a] | ........ | 41 800 | 69 900 | ..... | ........ | ........ | 34 050[b] | 79 800[b] | 0.815 |
| Nickel steel[a] | ........ | 48 000 | 91 400 | ..... | ........ | ........ | 41 700[b] | 91 400[b] | 0.869 |

[a] Annealed.

[b] Solid torsion specimen; all others slotted plate specimens.

[c] All determinations were made on solid torsion specimens.

[d] Not observed.

The ten alloy steels included manganese, nickel, chromium, molybdenum, vanadium and tungsten steels. Nine were tested in the form of $\frac{7}{8}$-in. diameter specimens in annealed and quenched-and-drawn conditions. The tenth was tested in the "as-rolled" condition using a $\frac{5}{8}$-in. diameter specimen. Chemical compositions and heat treatments are given in Table II.

The Bethlehem Steel Co., Bethlehem, Pa., supplied all materials for this investigation, with the exception of one of the alloy steels which was furnished by The Lukens Steel Co., Coatesville, Pa.

## SPECIMENS, METHODS AND APPARATUS

Specimens from the I-beams were taken in all cases from the webs. Since these were in most cases only about $\frac{3}{8}$ in. thick and the plates only about $\frac{1}{4}$ in. thick, the usual cylindrical torsion specimen could not be used for determining shearing properties and slotted plate specimens described below were used. Plates S-1 and S-2 were $\frac{5}{8}$ in. thick and both slotted plates and solid torsion specimens were made from these plates. For the remaining structural material and for all the alloy steels, shearing properties were determined on cylindrical torsion specimens. For reasons of economy, solid specimens were used, these being considerably less expensive than the hollow cylindrical specimens; however, both solid and hollow specimens were used in testing three grades of steel, designated soft, medium and hard in Table I. The tubular specimens were 1 in. in outside diameter with $\frac{1}{8}$-in. wall thickness.

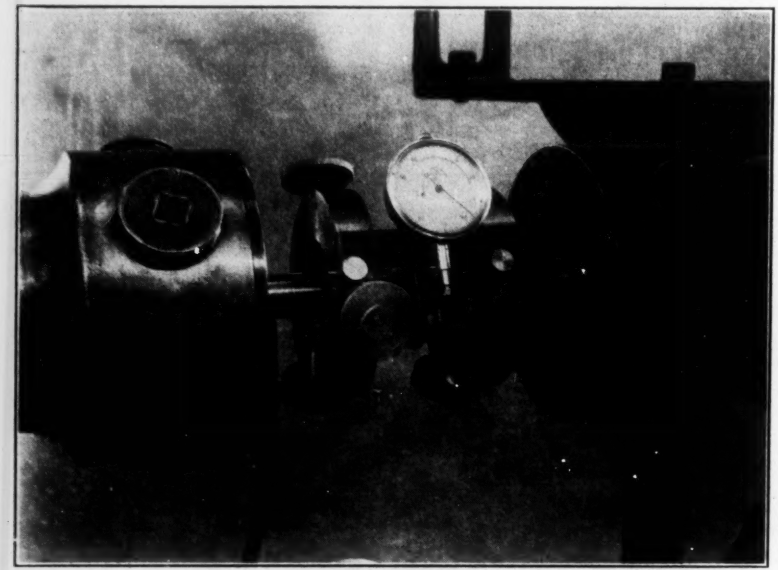

FIG. 1.—Apparatus for Measuring Torsional Deformations.

Tension and shearing tests were also made on $\frac{1}{2}$- and 1-in. diameter bars machined from a rolled $1\frac{1}{2}$-in. bar of structural steel, to determine any effect of size of specimen.

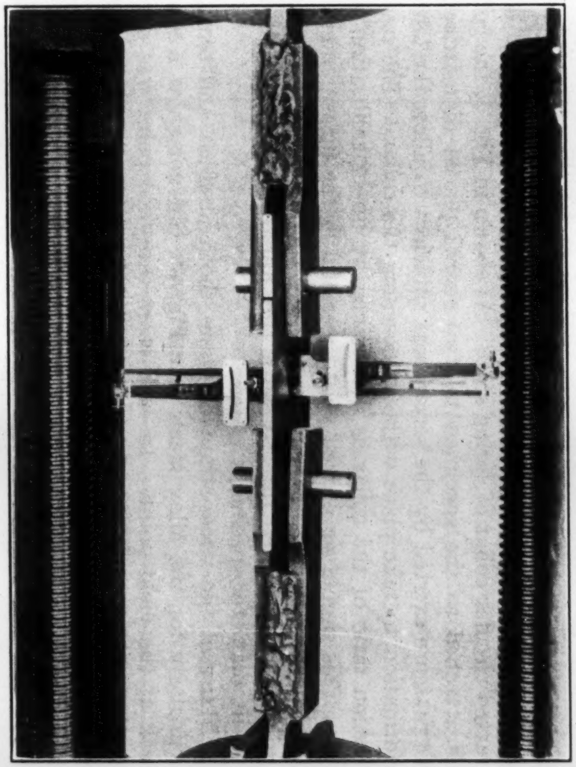

FIG. 2.—Slotted Plate Shear Specimen with Huggenberger Tensometer for Determination of Yield Point.

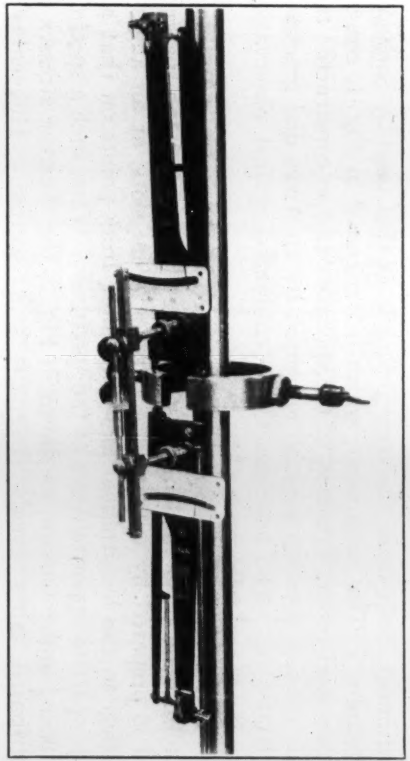

FIG. 3.—Apparatus for Measuring Lateral Deformations.

The shearing properties on cylindrical specimens were determined in a 24,000 in-lb. capacity Olsen torsion machine. The deformations were observed by means of a specially constructed instrument, built upon the same principle as that used by Seely and Putnam(**6**).[1] The instrument, which is illustrated in Fig. 1, consists of two steel collars attached to the specimen and so constructed that their relative rotation may be measured by an Ames dial graduated to 0.0001 in. The instrument was calibrated against observations by mirrors and telescopes.

The slotted plate specimen, first used by Moore and Wilson(**5**), is 4 in. wide by 9 in. long, of the same thickness as the material, and is prepared by cutting two parallel $\frac{1}{16}$-in. slots, at an angle of 45 deg. to the longitudinal axis, extending from points on that axis $\frac{1}{2}$ in. apart to opposite edges of the specimen. When such a specimen is placed under tension, as shown in Fig. 2, the shearing stresses are distributed fairly evenly over the $\frac{1}{2}$-in. section. Huggenberger tensometers were used for measuring deformations over $\frac{1}{2}$-in. gage lengths from which yield point in shear was determined, but not modulus of elasticity. Ultimate strength in shear was also determined on these specimens.

Tensile deformation was measured by a Ewing extensometer. In determining Poisson's ratio, lateral deformation was measured by means of Huggenberger tensometers attached to a specially constructed circular collar of spring steel. As shown in Fig. 3, the collar was attached to the specimen by two screws placed diametrically. The pressure exerted by the screws was sufficient to allow the lateral deformation to take place without loosening the collar. The multiplication ratio of the collar as determined by direct calibration was 2.16. This simple rig proved very successful in the measurement of lateral deformations both below and above the yield point. Lateral strains were measured directly with this apparatus to 0.000005 in. for 1-in. diameter specimens.

Many of the stress-strain diagrams, both in shear and tension, particularly for the alloy steels (see Fig. 5), did not show a sharp break at the yield point. In such cases the yield point was determined as the stress at which the rate of deformation is 100 per cent greater than the initial rate of deformation, that is, the stress at which the tangent to the stress-strain curve has a slope (ratio of stress to strain) one-half of the slope of the initial straight-line portion.

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[1] The boldface numbers in parentheses refer to the reports and papers given in the bibliography appended to this paper, see p. 284.

## RESULTS OF TESTS

The results of the tests on structural steels are given in Table I, and those on alloy steels in Tables II, III and IV. Typical stress-strain curves are shown in Figs. 4 and 5.

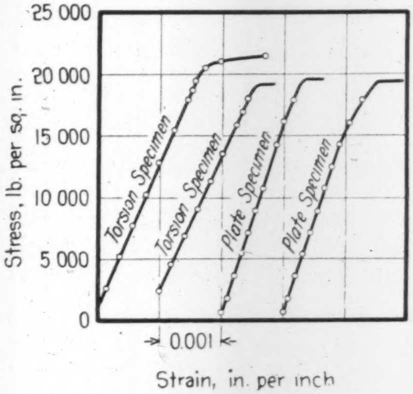

FIG. 4.—Relation Between Yield-Point Stresses as Determined on Solid Torsion and Slotted Plate Specimens.

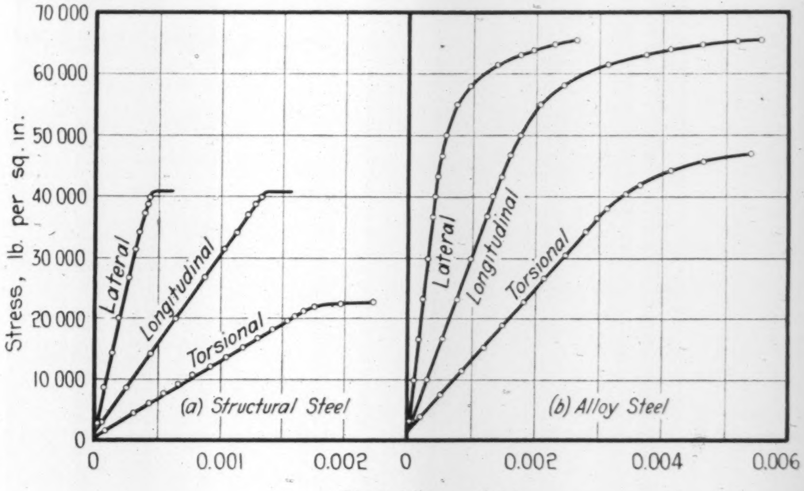

FIG. 5.—Typical Stress-Strain Diagrams.

### *Effect of Shape and Size of Test Specimen:*

Comparative stress-strain curves in shear obtained with slotted plate specimens and solid cylindrical specimens from the same material (plates S-1 and S-2) are shown in Fig. 4. It is noted that these two types of specimens gave very nearly the same yield point in shear. However, at a given stress the measured strains of the slotted plate specimens were less than those for the solid torsion bars. The slotted plate specimens gave a more definite yield point than did the torsion bars, and in general the results were more consistent.

TABLE II.—CHEMICAL COMPOSITION AND HEAT TREATMENT OF ALLOY STEELS.

| Steel | Chemical Analysis, per cent | | | | | | | | | | Treatment[a] |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Carbon | Manganese | Phosphorus | Sulfur | Silicon | Nickel | Chromium | Vanadium | Molybdenum | Tungsten | |
| No. 1.... | 0.27 | 0.72 | 0.014 | 0.024 | 0.21 | .... | .... | .... | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1600 F. (870 C.) water quenched, drawn to 1100 F. (595 C.) |
| No. 2.... | 0.38 | 0.65 | 0.015 | 0.029 | 0.22 | .... | .... | .... | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1600 F. (870 C.), water quenched, drawn to 1100 F. (595 C.) |
| No. 3.... | 0.43 | 0.64 | 0.015 | 0.023 | 0.20 | 3.47 | .... | .... | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1450 F. (790 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 4.... | 0.40 | 0.51 | 0.019 | 0.028 | 0.20 | 1.65 | 0.99 | .... | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1530 F. (830 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 5.... | 0.32 | 0.60 | 0.014 | 0.019 | 0.16 | 1.92 | 0.86 | .... | 0.30 | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1530 F. (830 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 6.... | 0.31 | 1.66 | 0.015 | 0.024 | 0.25 | .... | .... | .... | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1525 F. (830 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 7.... | 0.34 | 0.92 | 0.032 | 0.033 | 0.20 | .... | 0.51 | .... | .... | 1.20 | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1600 F. (870 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 8.... | 0.37 | 0.71 | 0.024 | 0.037 | 0.33 | .... | 1.18 | 0.16 | .... | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1600 F. (870 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 9.... | 0.33 | 0.54 | 0.025 | 0.029 | 0.21 | .... | 0.78 | .... | 0.24 | .... | A—Heated to 1450 F. (790 C.), furnace cooled<br>Q—Heated to 1600 F. (870 C.), oil quenched, drawn to 1100 F. (595 C.) |
| No. 10 .. | 0.20 | 1.19 | 0.033 | 0.019 | 0.67 | .... | 0.45 | .... | .... | .... | As rolled |

[a] A = annealed. Q = quenched and drawn.

The ultimate shearing strength was less when determined on a plate specimen, as reported in Table I, than when determined on a torsion bar, and the fracture of the former indicated a pure shear failure.

The average yield points in shear as determined on hollow and solid cylindrical specimens for the soft, medium and hard steels listed in Table I were as follows:

| | YIELD POINT, LB. PER SQ. IN. | | RATIO, |
|---|---|---|---|
| | HOLLOW SPECIMEN | SOLID SPECIMEN | YIELD POINT HOLLOW TO YIELD POINT SOLID |
| Soft steel | 19 650 | 20 200 | 0.972 |
| Medium steel | 20 600 | 22 750 | 0.906 |
| Hard steel | 27 950 | 34 050 | 0.820 |

It will be seen that the yield point in shear determined on the hollow specimens averaged about 90 per cent of that determined on solid bars.

The tests to determine effect of size of specimen show that the smaller specimens (½ and 1 in. in diameter) machined from 1½-in. bars gave lower strengths in both tension and shear than did the 1½-in. specimens, due probably to the effects of rolling during manufacture. The moduli of elasticity and Poisson's ratio, however, were very nearly the same for the several sizes of specimens. (See Table I.)

*Properties of Structural Steels:*

Referring to Table I, the shearing yield point for ordinary structural steels varied from 16,500 to 29,200 lb. per sq. in.; the yield point in tension varied from 26,900 to 51,300 lb. per sq. in. for the same materials. Steels containing nickel gave shearing yield point as high as 41,700 lb. per sq. in.

The ratios between the yield points in shear and in tension, given in the last column of the table, varied greatly. For rolled I-beams, the ratio varied from 0.505 to 0.634; for tank steel plates, from 0.509 to 0.534; for rolled soft steel plates, from 0.527 to 0.671; and for rolled 1½-in. bars the average ratio was 0.629. It should be noted that a number of the structural grade steels had shearing yield points, when determined on slotted plate specimens, considerably less than 60 per cent of the yield point in tension. If the stress developed by a hollow torsion specimen is considered to be correct, these ratios would be correspondingly lower.

The addition of nickel raised the ratio of yield points in shear and in tension to as much as 0.869.

Poisson's ratio for the different types of structural steel varied only between 0.271 and 0.302. For design purposes, the use of a value of 0.3 seems to be justified.

The shearing modulus of elasticity showed a high degree of uniformity when calculated from the observed longitudinal and lateral strains. However, the modulus obtained by direct measurements on the torsion specimens showed considerable variation.

Typical stress-strain diagrams for longitudinal, lateral and torsional deformations of structural steel are shown in Fig. 5. It is noted that the lateral deformation indicated a sharp yield point at the same stress as did the longitudinal deformation.

TABLE III.—HARDNESS AND DUCTILITY OF ALLOY STEELS.

The values represent the average of two determinations.

| Steel[a] | | Brinell Hardness Number | Elongation in 2 in., per cent | Reduction of Area, per cent | Appearance of Tension Test Fracture |
|---|---|---|---|---|---|
| No. 1 | A | 153 | 46.5 | 64.0 | Cup |
| | Q | 191 | 42.0 | 70.5 | Star |
| No. 2 | A | 146 | 44.5 | 55.6 | Cup, medium grained |
| | Q | 204 | 40.0 | 65.3 | Star, finely grained |
| No. 3 | A | 187 | 33.0 | 44.7 | Cup, finely grained |
| | Q | 226 | 33.5 | 58.2 | Star, very fine grain |
| No. 4 | A | 170 | 39.5 | 54.5 | Irregular cup, finely grained |
| | Q | 224 | 34.0 | 59.2 | Ruptured, badly laminated |
| No. 5 | A | 202 | 37.3 | 57.6 | Semi-cup, finely grained |
| | Q | 229 | 32.0 | 63.7 | Irregular star, badly laminated |
| No. 6 | A | 169 | 42.0 | 54.5 | Cup, finely grained |
| | Q | 204 | 33.0 | 57.0 | Step, laminated |
| No. 7 | A | 185 | 40.0 | 56.0 | Cup, finely grained |
| | Q | 285 | 27.0 | 52.9 | Star, laminated |
| No. 8 | A | 179 | 41.5 | 62.1 | Star, finely grained |
| | Q | 285 | 24.5 | 48.5 | Star, slightly laminated |
| No. 9 | A | 170 | 48.0 | 66.0 | Cup, finely grained |
| | Q | 229 | 28.0 | 59.5 | Star, finely grained |
| No. 10[b] | | 156 | 30.3 | .... | Square, laminated |

[a] A = annealed. Q = quenched and drawn.

[b] As rolled.

*Properties of Alloy Steels:*

The hardness and ductility of the alloy steels are given in Table III, together with a description of the tension test fracture. Attention is called to the laminated fractures of the quenched-and-drawn specimens.

Tensile and shearing properties and Poisson's ratio are presented in Table IV. The yield point in tension varied from 37,500 to 53,000 lb. per sq. in. for the steels in the annealed condition, and from 55,000 to 124,000 lb. per sq. in. for those in the quenched condition. The yield point in shear as determined on solid torsion specimens varied from 33,500 to 43,750 lb. per sq. in. for the annealed condition and from 44,100 to 93,000 lb. per sq. in. for the quenched condition. In general, the ratios between the yield points in shear and in tension were greater for the annealed than for the quenched specimens, ranging from 0.774 to 0.894 for the former and from 0.662 to 0.809 for the latter. The alloy steels, both in annealed and quenched conditions, showed considerably greater ratios between yield points in shear and tension than the structural steels.

TABLE IV.—TENSILE AND SHEARING PROPERTIES AND POISSON'S RATIO FOR ALLOY STEELS.

The values represent the average of two determinations.

| Steel[a] | | Tension | | | Poisson's Ratio, Observed | Shear[b] | | | | Ratio, Yield Point in Shear to Yield Point in Tension |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Modulus of Elasticity, lb. per sq. in. | Yield Point, lb. per sq. in. | Ultimate Strength, lb. per sq. in. | | Modulus of Elasticity, lb. per sq. in. | | Yield Point, lb. per sq. in. | Ultimate Strength, lb. per sq. in. | |
| | | | | | | Observed | Calculated[c] | | | |
| No. 1 | A | 27 500 000 | 37 500 | 67 400 | 0.316 | 11 780 000 | 10 460 000 | 33 500 | 75 900 | 0.894 |
| | Q | 29 600 000 | 55 000 | 91 100 | 0.310 | 11 975 000 | 11 300 000 | 44 100 | 90 600 | 0.803 |
| No. 2 | A | 28 750 000 | 41 500 | 75 700 | 0.287 | 11 670 000 | 11 180 000 | 36 200 | 79 800 | 0.872 |
| | Q | 30 000 000 | 57 750 | 99 500 | 0.296 | 12 380 000 | 11 580 000 | 46 650 | 96 700 | 0.809 |
| No. 3 | A | 30 000 000 | 53 000 | 94 400 | 0.308 | 12 110 000 | 11 500 000 | 41 500 | 90 600 | 0.784 |
| | Q | 28 200 000 | 82 200 | 117 000 | 0.298 | 11 660 000 | 10 880 000 | 64 750 | 106 800 | 0.787 |
| No. 4 | A | 28 800 000 | 43 750 | 89 900 | 0.299 | 11 320 000 | 11 100 000 | 33 800 | 90 600 | 0.774 |
| | Q | 29 050 000 | 91 000 | 129 150 | 0.290 | 12 085 000 | 11 280 000 | 66 500 | 115 000 | 0.730 |
| No. 5 | A | 28 700 000 | 49 600 | 96 150 | 0.288 | 11 500 000 | 11 150 000 | 40 000 | 95 750 | 0.805 |
| | Q | 28 500 000 | 105 000 | 140 000 | 0.285 | 11 700 000 | 11 100 000 | 69 500 | 122 350 | 0.662 |
| No. 6 | A | 27 850 000 | 43 250 | 84 900 | 0.295 | 12 000 000 | 10 770 000 | 36 000 | 84 300 | 0.831 |
| | Q | 28 650 000 | 78 500 | 112 000 | 0.296 | 12 095 000 | 11 080 000 | 58 000 | 100 650 | 0.740 |
| No. 7 | A | 28 500 000 | 48 000 | 82 500 | 0.320 | 12 700 000 | 10 800 000 | 42 000 | 84 850 | 0.875 |
| | Q | 29 400 000 | ...... | 157 500 | 0.281 | 12 165 000 | 11 470 000 | 90 000 | 134 950 | ..... |
| No. 8 | A | 29 350 000 | 49 250 | 88 700 | 0.289 | 11 750 000 | 11 400 000 | 43 750 | 89 500 | 0.889 |
| | Q | 30 000 000 | 124 000 | 151 350 | 0.279 | 12 500 000 | 11 750 000 | 93 000 | 131 650 | 0.750 |
| No. 9 | A | 28 550 000 | 42 500 | 76 700 | 0.288 | 12 000 000 | 11 100 000 | 35 000 | 87 350 | 0.824 |
| | Q | 28 750 000 | 90 500 | 126 500 | 0.272 | 11 845 000 | 11 300 000 | 63 750 | 114 000 | 0.705 |
| No. 10[d] | | 30 425 000 | 54 300 | 85 600 | 0.276 | 11 875 000 | 11 920 000 | 31 100 | 84 200 | 0.573 |

[a] A = annealed. Q = quenched and drawn.
[b] Determined on solid torsion specimens.
[c] Calculated from observed tension modulus of elasticity and Poisson's ratio.
[d] As rolled.

A number of the annealed solid torsion specimens gave ultimate strengths in shear in excess of the corresponding tensile strengths. This is due to the use of the ordinary torsion formula to determine ultimate strength, notwithstanding that the formula does not hold after the stress has exceeded the proportional limit of the material.

Poisson's ratio varied from 0.272 to 0.320 for these ten alloy steels. With the exception of the tungsten steel (No. 7) there was little difference between Poisson's ratio for the steel in the annealed and in the quenched conditions.

Typical stress-strain diagrams for longitudinal, lateral and torsional deformations of one of the alloy steels are shown in Fig. 5. Attention is called to the gradual transition from a straight line to a curve in all three cases. After the proportional limit was reached, the relative rate of lateral deformation increased over that of the longitudinal deformation so that Poisson's ratio approached 0.5.

## SUMMARY

The following summary and conclusions may be presented from this investigation:

1. The apparatus developed for measuring lateral and torsional strains proved very successful, based on accuracy and consistency of results and calibration against other instruments.
2. Slotted plate specimens and solid torsion specimens gave approximately the same yield point in shear.
3. The average yield point in shear of a hollow bar was about 90 per cent of that of a solid bar.
4. The size of the test specimen had no effect upon the observed Poisson's ratio.
5. Steel of structural grade showed a wide variation in the ratio of yield point in shear to yield point in tension. In general the ratio was below 0.60 for specimens from rolled plates and structural sections.
6. Poisson's ratio for structural steel varied from 0.271 to 0.302.
7. For alloy steels the ratio of yield points in shear and in tension varied between 0.774 and 0.894 for the annealed condition and between 0.662 and 0.809 for quenched-and-drawn condition.
8. Poisson's ratio for alloy steels varied between 0.272 and 0.320.

---

## BIBLIOGRAPHY

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Yield point in shear found to be approximately 60 per cent of that in tension.

(2) L. B. Turner, "The Elastic Breakdown of Materials Submitted to Compound Stresses," *Engineering* (London), p. 203, February 12, 1909.

(3) L. B. Turner, "The Strength of Steels in Compound Stress and Endurance Under Repetition of Stress," *Engineering* (London), p. 115, July 28, 1911.

Yield point in shear found to vary between 50 and 58 per cent of that in tension.

(4) E. L. Hancock, "Results of Tests of Materials Subjected to Combined Stresses," *Proceedings*, Am. Soc. Testing Mats., Vol. VIII, p. 373 (1908).

Ratios of yield points in shear and in tension varied from 0.45 to 0.69.

(5) H. F. Moore and W. M. Wilson, "The Strength of Webs of I-Beams and Girders," *Bulletin 86*, Engineering Experiment Station, University of Illinois, May, 1916.

Reported ratios of yield points in shear and in tension varied between 0.61 and 0.71. Slotted plates were used as shear specimens.

(6) F. B. Seely and W. J. Putnam, "The Relation Between the Elastic Strengths of Steel in Tension, Compression and Shear," *Bulletin 115*, Engineering Experiment Station, University of Illinois, November, 1919.

The proportional limit in shear ranged from 53 to 64 per cent of that in tension for hollow specimens, and from 66 to 80 per cent for solid specimens of different steels.

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Report on effect of form and size of specimen upon results of torsion tests.

## (*b*) POISSON'S RATIO

(8) J. Bauschinger, "Über die Quercontraction und Dilatation bei der Längenausdehnung und Zusammendrückung Prismatischer Körper," *Der Civilingenieur*, Vol. XXV, p. 81 (1879).

Poisson's ratio approximately 0.29 for structural steel.

(9) C. E. Stromeyer, "Experimental Determination of Poisson's Ratio," *Proceedings*, Royal Soc. (London), Vol. LV, p. 373 (1894).

Poisson's ratio varied from 0.27 to 0.30 for iron and steel.

(10) J. Morrow, "On an Instrument for Measuring the Lateral Contraction of Tie-Bars and on the Determination of Poisson's Ratio," *Proceedings*, Physical Soc. (London), Vol. XVIII, p. 582 (1901-1903).

Poisson's ratio for mild steel varied from 0.27 to 0.28.

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Poisson's ratio equal to 0.30 for annealed and 0.27 for as rolled structural steel. Checked elastic relation between modulus of elasticity in shear and in tension and Poisson's ratio.

(12) T. McLean Jasper, "Determination of Poisson's Ratio and a Suggestion for Its Use in Stress Analysis," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 1012 (1924).

Values of Poisson's ratio ranged from 0.20 to 0.27 for different steels.

# DISCUSSION

Mr. F. B. Seely.[1]—In the stress-strain diagram, in getting the yield point, the curve seems to run above the highest plotted point and then break over. I wonder why that was done; was there another point not shown in the diagram? It seemed to raise the yield point somewhat. Another question: was any arbitrary method of determining the yield point other than the 100 per cent increase in the slope used? Might not the ratio of yield points be different if some other arbitrary method of selecting the so-called yield point were used? Then another point: the question might be raised as to whether it is possible to determine the torsional shearing yield point without the use of a hollow specimen.

The results reported on yield point in shear check fairly well with the tests made at the University of Illinois a few years ago, although more of our work was done on hollow specimens.

Mr. H. J. Godfrey.[2]—In answer to Mr. Seely's questions, the reason that there is, in some cases, no point plotted at the yield point of the material, is the fact that the yield point was determined by the sudden movement of the pointer on the extensometer. Only one arbitrary method was used in the determination of the yield point; if another method had been used I do not believe that the ratio between the shearing and tensile yield points would have been much different. However, if the yield point as determined on the hollow torsion specimens had been used, the ratios would have been changed.

In amplification of the discussion in the paper of the slotted plate specimen, we have made photoelastic studies which show very well the stress distribution. The shearing stress is shown to be fairly uniform along the section under stress. There are some concentrated stresses at the ends of the slots but these should not interfere with the section under shearing stress.

Mr. H. F. Moore.[3]—I was very much interested in the care Mr. Lyse took in getting his Poisson's ratio by two methods: (1) direct measurement, which is the most obvious method and which involves very delicate methods of measuring lateral strain, and (2) by comparison of moduli of elasticity in tension, and in torsion, which seems a simple method but is very deceptive in that a slight error in determining either modulus introduces a much larger error in Poisson's ratio.

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[1] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Research Fellow in Civil Engineering, Lehigh University, Bethlehem, Pa.

[3] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

It is quite necessary that the strain measuring apparatus and load measuring apparatus in the two cases be very carefully cross calibrated. Correctness of measurement makes no difference in ordinary tension tests, so long as the readings of the extensometer are *proportional* to strain, but in determining moduli correctness of extensometer reading *does* make a serious difference.

I beg leave to refer to some determinations of Poisson's ratio we have been making at Illinois during the past year, using a 10-in. steel shaft in compression, and using a 3,000,000-lb. machine to apply the compressive load, putting on extensometers for the longitudinal deformation and the lateral deformation and carefully cross calibrating those two extensometers against Johansson blocks. The estimated maximum error of strain reading was one or two one-hundred thousandths of an inch, and in measuring lateral expansion there was a 10-in. gage length. The values obtained were slightly higher than reported in the paper, running, I think, from 0.300 to 0.320. However, it should be remembered that the specimen was in compression rather than in tension.

MR. T. MCL. JASPER.[1]—A few years ago some determinations of Poisson's ratio were made by measuring directly the lateral and the longitudinal deformation and also by measuring the elastic properties and calculating the values of Poisson's ratio. It was very evident that a cold-rolled steel indicated a relatively small value of Poisson's ratio in tension, and I have since thought that, had we measured Poisson's ratio on the cold-rolled material in compression we might have had a relatively large value of Poisson's ratio.

MR. A. NADAI[2] (*by letter*).—The authors compare stress-strain curves obtained from ordinary tension tests with diagrams constructed from torsion tests with round bars. The stresses in these diagrams (applying to the plastic range) were computed by means of the ordinary formulas, assuming a proportionality between strains and stress. The writer believes that it would have perhaps been not much more troublesome to construct diagrams in the plastic region from their torsion tests made with the round bars, in which the ordinates would have been the true maximum shearing stresses acting at the periphery of the circular cross-sections while the bars were yielding. All that would have been necessary would have been to compute the shearing stress from the relation

$$\tau_{max} = \frac{1}{2\pi a^3}\left(\theta \cdot \frac{dM}{d\theta} + 3M\right)$$

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[1] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[2] Research Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

where $2a$ is the diameter of the bar, $M$ is the torque and $\theta$ the angle of twist per unit of length and the function $M = F(\theta)$ can be determined empirically from the torsion test. The writer cannot see an advantage in the suggestion made in the paper to define a "yield stress" for shear taken from a torsion test of a round bar by drawing a tangent line inclined at an arbitrary angle to a curve, the ordinates of which were computed similarly in an arbitrary way (namely by using the proportionality of stress and strain in the plastic range, where it certainly is far from being observable). If the designer of heavy steel beams has good reasons to suspect that the shearing stresses may become large in the web of his beams, he will probably be anxious to know these stresses more exactly. It may, for example, occur that a "yield stress" cannot be detected by any means, simply because such a limiting stress at which plasticity starts does not exist for materials having certain types of stress-strain relations.

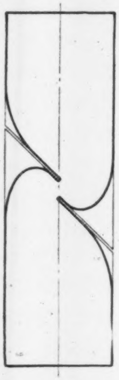

FIG. 1.—Test Specimen Showing Contour Line.

Another consequence which the designer may book to his advantage when computing the right stresses in his stress-strain diagrams would be that the ratio of the shearing strength (for yielding) to that for pure tension, he would observe, would have been found to be fairly constant (= 0.57) for a given material (a ductile metal), as has been observed by a number of more recent experimenters, such as W. Lode, Quinney and G. T. Taylor, Ros and Eichinger.

From a photoelastic study recently made at the Westinghouse Research Laboratories, it can be concluded that the maximum shearing stresses along the axis of the slotted specimen are far from being uniformly distributed and tend to increase considerably towards the re-entrant corners of the slots. As the four projecting corners at the beginning of the slots are inactive regions in the bar, through which not much stress is transmitted, these regions could be cut off without disturbing greatly the stress distribution in the remaining portions of the bar. This has been indicated by the thick contour line in the accompanying Fig. 1. The slotted specimen can therefore be regarded in first approximation as a tension test specimen having the shape indicated: a bar having a variable width, a curved central line (variable eccentricities) and two sharp re-entrant corners.

MR. C. W. MACGREGOR[1] (*by letter*).—A number of tests are described in the paper in which the yield points and ultimate strengths of plate material were determined by means of a slotted test specimen. It is stated in the paper that when such a specimen is placed under tension, the shearing stresses are distributed "fairly uniformly" over the ½-in. section. At the suggestion of Mr. Nadai, of the Westinghouse Research Laboratories, the writer has made a photoelastic test in order to determine the actual shear stress distribution over this ½-in. section between the slots, using a bar of transparent bakelite similar to the slotted steel bars described in the paper. The axis of the test specimen passed through the center of the ends of the slots, and the corners of these slots were left sharp.[2]

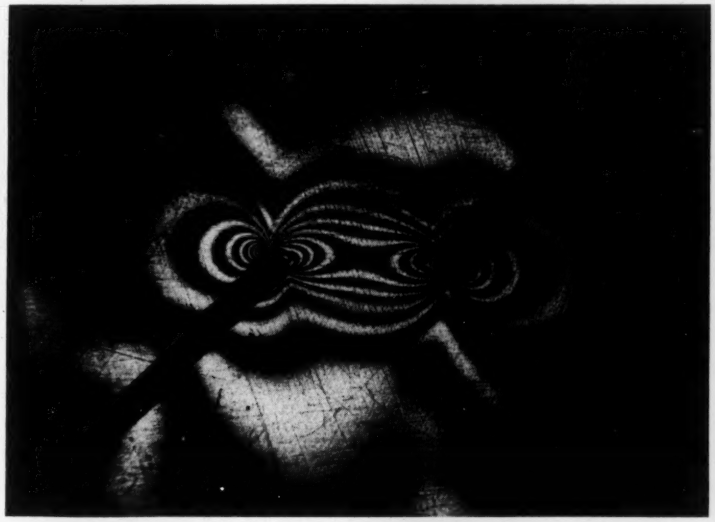

FIG. 2.—Isochromatics Obtained by Loading a Slotted Bakelite Test Specimen.

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[1] Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa

[2] Although a drawing of the details of the slots near the ½-in. section was not contained in the paper itself, the fact whether the inner corners of the slots in the steel test specimens were sharp or rounded will not greatly affect the results obtained from the photoelastic test with the exception of a region around the corners themselves where a secondary effect of stress concentration will be produced in case of sharp corners.

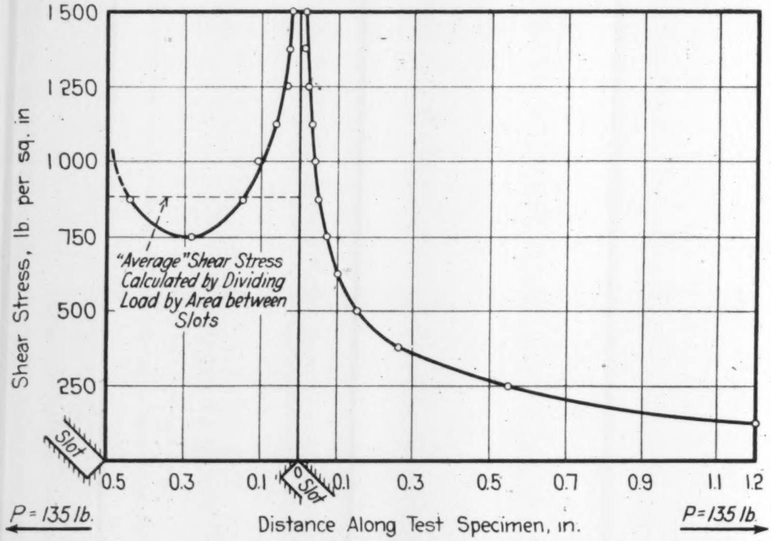

FIG. 3.—Shear Stress Distribution Along Line Parallel to Center Line of Slotted Bar Joining Corners of Slots, Determined by Photoelastic Test (Load, $P$ = 135 lb.).

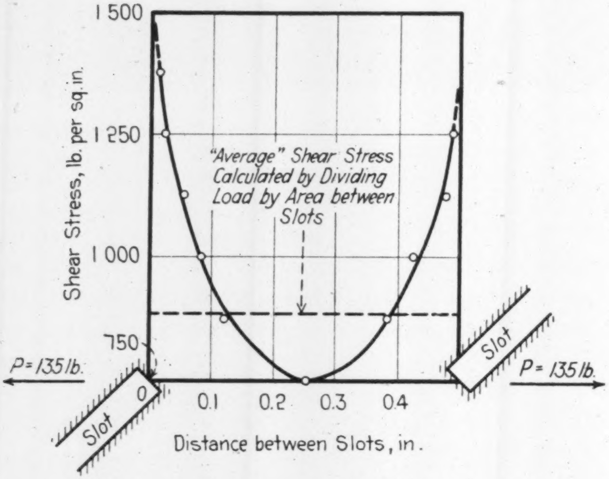

FIG. 4.—Shear Stress Distribution Along Line Joining Centers of Slots Determined from Photoelastic Test (Load, $P$ = 135 lb.).

The accompanying Fig. 2 shows the slotted region of the test specimen near the ½-in. section under a tensile load of 135 lb. The dark bands visible in the photograph represent the so-called "isochromatics," or lines along which the maximum or principal shearing stress has a constant value. For this material, the portion of the test specimen covered by one fringe possesses a maximum shearing stress which is 125 lb. per sq. in. different in value from that in the part of the bar covered by an adjacent band. Hence, it is readily apparent from Fig. 2 that since a considerable number of different fringes cross the ½-in. section between the slots the shearing stress along this section is far from being uniform. To show the actual distribution at this same load of 135 lb., the maximum shearing stress was plotted in Figs. 3 and 4, respectively. Figure 3 shows the variation of the maximum shearing stress along the line parallel to the longitudinal axis of the bar passing through the corners of the two slots; Fig. 4 represents the shear stress distribution along the axis of the bar between the slots. In each figure the "average" shearing stress calculated by dividing 135 lb. by the ½-in. sectional area is indicated for comparison. It is to be noticed from the figures that the maximum shearing stress varies from values above 1500 lb. per sq. in. to about 750 lb. per sq. in. and reaches still higher values near to the sharp corners.

Judging then from the character of this variable distribution of shearing stress along the bar axis, it appears rather difficult to determine correctly such sensitive physical properties as the yield point in pure shear by means of this type of test. Due to the high stress concentration at the ends of the slots, local yielding will furthermore begin there at a much lower load than that for which the entire section yields.

MESSRS. INGE LYSE[1] AND GODFREY (*authors' closure, by letter*).—The authors cannot agree with Mr. Nadai regarding the importance of the plastic flow in torsion. They believe every structure should be so designed that a proper margin of safety, based on yield-point stress, is always present. Mr. Timoshenko[2] states: "In the case of structural steel, it is logical to take the yield point as the basis for calculating the working stress because here a considerable permanent set may occur, which is not permitted in engineering structures." As stated in the paper, the yield points of the steel in shear and in tension were the important items investigated in this study. The ordinary simple torsion formula holds up to the yield point, so that the authors saw no reason for introducing the complicated type of formula presented by Mr. Nadai. The "useful yield-point stress" can be determined just as accurately and more conveniently by the use of the diagram based on the straight-line than by Mr. Nadai's formula. The authors do not see any practical value in a determination of the stresses beyond the yield point because here the structure deforms beyond the limit of usefulness. It was the yielding of steel beams (in tests conducted at the Fritz Engineering Laboratory) beyond the limit of usefulness at stresses corresponding to the yield-point stress in shear of the material that led to this investigation.

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[1] Research Associate Professor of Engineering Materials, Lehigh University, Bethlehem, Pa.

[2] S. Timoshenko, "Strength of Materials," Part I, p. 8 (1930).

Mr. Nadai also states that for ductile materials the yield point in shear is 57 per cent of that in tension, which is in support of the maximum shear-strain theory of failure. He refers to tests by a few experimenters who have obtained values near the given ratio, neglecting all other experiments. The authors subscribe to the conclusion reached by Mr. Salmon[1] after he had made a complete review of the different theories of failure: "It will be apparent from the preceding evidence that the correctness of any one of the theories outlined above is yet to be definitely established, and that the criterion for elastic breakdown is still an open question."

The photoelastic reproduction presented by Mr. MacGregor is very similar to that obtained by the authors. Although the stress was not uniform across the section under load, the effect upon the yield point was negligible, as shown in Fig. 4 of the paper. For ductile materials which were used exclusively in the plate specimens, a concentration of stress does not materially affect the yield point. There is therefore no cause for anxiety from the fact that the photoelastic study shows stress concentrations at low loads. Mr. Salmon states: "The stress distributions determined by analysis, and by the polarized light or soap-film methods, only hold while the material remains elastic. When in ductile material, at a stress concentration, the proportional limit and yield point are passed flow begins and a redistribution of stress takes place, accompanied by a certain amount of strain hardening, mitigating to a large extent the unfavorable stress distribution." The results showed that such a redistribution of stress took place, bringing the yield point of slotted plates nearly equal to that of torsion bars, indicating that without the proper realization of the property of the material, one might easily be misled by purely theoretical deductions and photoelastic stress analysis. Hence the importance of practical judgment in engineering materials.

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[1] E. H. Salmon, "Materials and Structures," p. 498, London (1931).

# STUDIES ON A MODIFICATION OF THE ROHN TEST FOR INVESTIGATING CREEP OF METALS

By C. R. Austin[1] and J. R. Gier[2]

## Introduction

Work is in progress in the Westinghouse Research Laboratories under the direction of Messrs. Nadai[3] and McVetty[4] whose object is to express the general conditions affecting working stresses of metals at elevated temperatures and the creep behavior of metals by general mechanical means. The work here reported is not intended to cover this field, but rather to discuss our experiences with a particular test method that appeared to offer possibilities as a rapid means for classifying alloys as to their metallurgical behavior and probable utility for use in mechanical service at high temperatures.

These results are not offered as a means for quantitative evaluation of creep characteristics.

While the value of creep data as furnished by the conventional creep test is undeniable from the point of view of the design engineer, the use of the conventional creep test as a means of classifying alloys is both expensive and extremely slow. It is on this account that numerous attempts have been made to devise some form of short-time test which would give reliable data. An excellent summary on this phase of the subject is given by Tapsell.[5] Data obtained from a form of bend test have also shown promise as providing a "merit index" of high-temperature serviceability.[6]

The present paper deals with our experiences with a refinement of the Rohn type of creep test and presents some data on pure iron that exemplifies the behavior of the apparatus we have used.

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[1] Section Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Associated with Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[3] A. Nadai, "The Creep of Metals," The New Haven Meeting of A.S.M.E. Applied Mechanics Division, June, 1931.

[4] P. G. McVetty, "Factors Affecting Choice of Working Stresses for High Temperature Service," *Transactions*, Am. Soc. Mechanical Engrs., December, 1932.

P. G. McVetty, "Testing of Metals at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 60 (1928).

[5] H. J. Tapsell, "Creep of Metals," Oxford University Press (1931).

[6] C. R. Austin and G. P. Halliwell, "Some Developments in High Temperature Alloys in the Nickel-Cobalt-Iron System," *Technical Publication 430*, Am. Inst. Mining and Metallurgical Engrs., September, 1931.

## ROHN TYPE OF TEST

The first account of this work was given by W. Rohn[1] in 1930 in a paper in honor of Dr. Wilhelm Heraeus. He described a method for testing metals at high temperatures which provides an automatic downward adjustment of temperature in response to creep in the test specimen as well as temperature regulation after the manner of a dilatation thermometer. The apparatus consists of a rod test specimen about one meter long contained in a vertical furnace having an approximately uniform temperature along its length. The test rod is put under a definite constant stress with a definite constant load. Since the rod is used as a sort of dilatation thermometer, it effects the automatic regulation of the temperature of the test furnace by means of control contacts actuated by changes in the length of the test rod.

Assume the temperature of the rod to be any temperature sufficiently high that creep takes place under the given load. This results in stretching the rod so that a contact fixed to its base breaks the electrical circuit and the temperature of the furnace falls to that point where the shrinkage of the rod due to fall in temperature compensates for the increase due to creep. If further creep takes place at this lower temperature there is a similar break in the electrical circuit whereby the test temperature again falls. A final limiting condition is approached asymptotically at which the temperature of the rod has been progressively reduced to that temperature where it is capable of maintaining the load without accepting further measurable deformation.

A continuous record may be taken of the time-temperature relationships. With temperature as ordinate and duration of test as abscissa it may readily be understood that the above record will show a fairly rapid fall of the curve in the early stages followed by a gradual flattening out toward a position parallel with the abscissa.

## TYPE OF TEST

In order that reliable data could be obtained from these tests it was considered essential that careful consideration be given to the following points:

1. The furnace should have a substantially uniform temperature over a considerable length, and between these limits and the ends of the furnace the fall in temperature should be as abrupt as possible. It could then be presumed that with a loaded test rod of uniform

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[1] W. Rohn, "Festschrift tum 70 Geburstag von Dr. Phil. Dr. Ing E. L. Wilhelm Hereaus in Hanau," reviewed in *The Metallurgist* (supplement to *The Engineer*), February 28, 1930, p. 22.

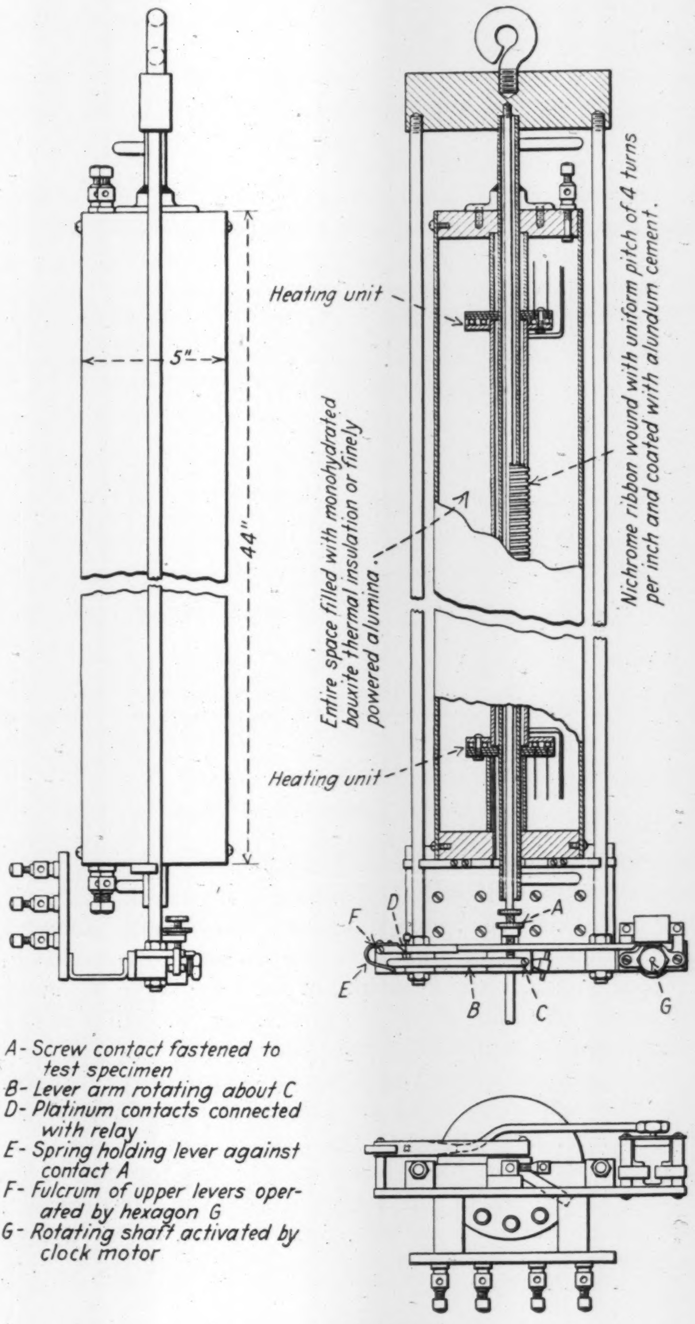
5"
44"
Heating unit
Heating unit
Entire space filled with monohydrated bauxite thermal insulation or finely powered alumina.
Nichrome ribbon wound with uniform pitch of 4 turns per inch and coated with alundum cement.
A
B
C
D
E
F
G
A- Screw contact fastened to test specimen
B- Lever arm rotating about C
D- Platinum contacts connected with relay
E- Spring holding lever against contact A
F- Fulcrum of upper levers operated by hexagon G
G- Rotating shaft activated by clock motor

FIG. 1.—Detail of Furnace.

diameter all significant creep would take place in the section within the uniform temperature range.

2. The test rod should not be subject to temperature fluctuations due to sudden marked line voltage changes or to cyclic fluctuations due to the lag between the length changes in the test rod and temperature changes in the furnace.

3. The test rod should not be subject to high-temperature corrosion and hence to marked local changes in cross-sectional area.

Considerable difficulty was encountered in constructing a vertical furnace about 3 ft. long which had any approximation of temperature uniformity over about 20 in. of its length. Several methods of construction received experimental consideration and the construction details finally employed in the last furnace built are shown in Fig. 1.

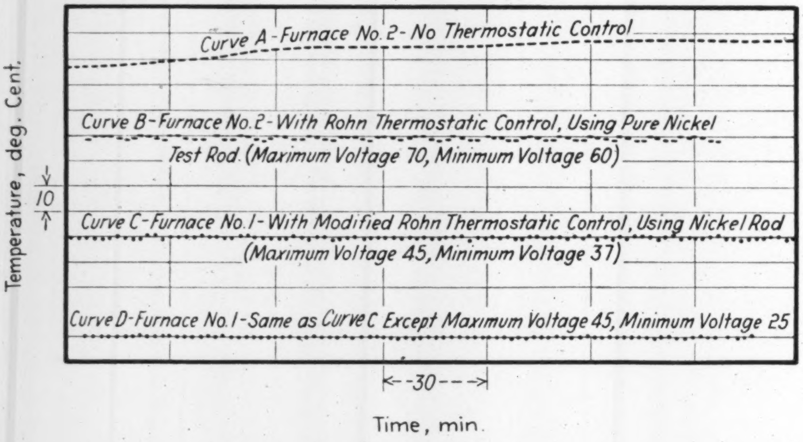

FIG. 2.—Temperature-Time Curves.

The 3 ft. long $1\frac{1}{8}$ in. bore silica tube was wound uniformly with nickel-chromium resistor ribbon. Independently controlled heating units placed at the top and bottom of the tube serve to compensate for end losses. An alloy steel tube passing up the center of the silica tube maintained a controlled atmosphere around the test rod which was screwed into the supporting block at the top of the furnace.

Into this supporting block are screwed the two Invar rods which hold the control mechanism at the base of the furnace in a fixed position relative to the block and hence to the top end of the test rod. On the lower end of the test rod is fixed the screw contactor, *A*. Any change in length of the rod, due to creep, or to expansion or contraction by heat, results in the contactor operating a lever arm, *B*, pivoted at *C* which results in a make or break at *D* of the relay circuit. The spring, *E*, keeps the lever in constant contact with *A*.

With the test set with a given load at some given elevated temperature, assuming that there is no creep, then the temperature of the test rod and furnace remains "constant." A slight temperature rise results in a slight increase in the length of the test specimen which depresses the lever *B* and breaks the platinum contacts at *D*, thus cutting off the current through the furnace windings. The resultant temperature reduction leads to a decrease in the length of the test rod, thus permitting the contacts to remake and restore the current to the heating circuits. Actually this make and break results in a shift from a maximum to a minimum current rather than to a complete shut-off of the power.

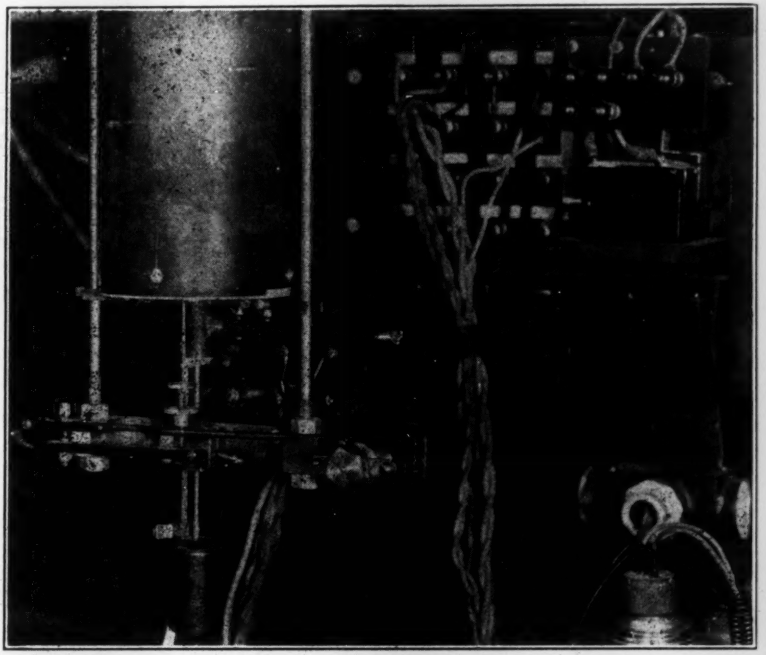

FIG. 3.—Control Apparatus.

If there were no lag between test and furnace temperature, a constant temperature would be maintained. This condition is obviously not realized experimentally, and a temperature-time record under the above conditions results in the familiar sine type of curve. Figure 2 illustrates this point. Curve *A* presents the temperature-time curve with the furnace operated directly on the line voltage, and curve *B* illustrates the type of curve just described.

Assuming now that creep takes place, the contact is broken and the temperature falls to such an extent that the amount of creep is exactly compensated for by the contraction of the rod due to the fall in temperature. Since small changes in temperature are considered to have marked effects on creep rates and since in the present test, temperature change is the dependent variable and is used to indicate the amount of creep in the test bar, it is essential that temperature fluctuations be reduced to a minimum.

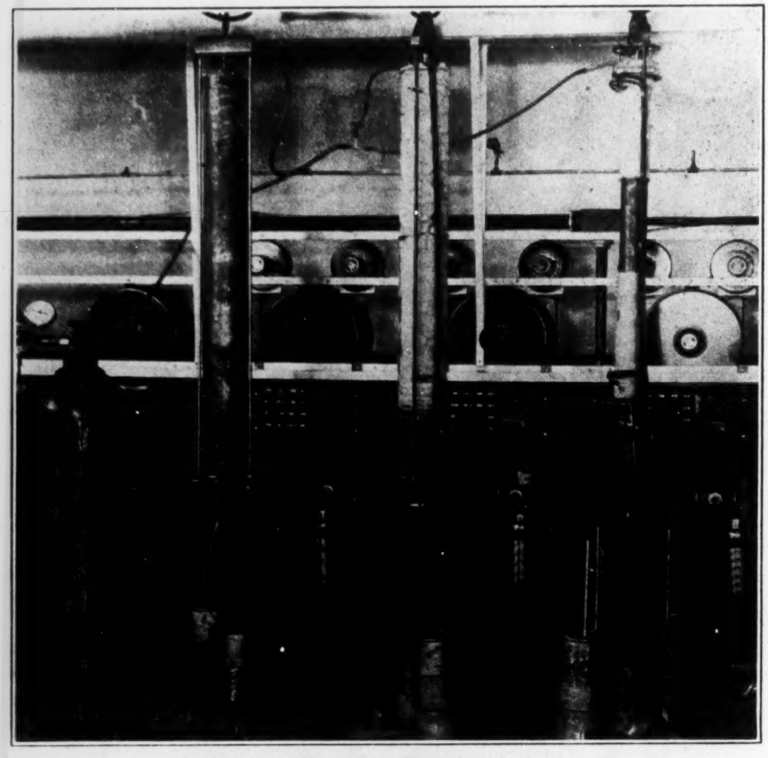

FIG. 4.—Complete Test Apparatus.

Data obtained in some of the early tests showed marked irregularities due to line voltage changes and temperature lag between furnace and specimen even with the aforementioned control. In the early stages of the test this was not so important, but when the temperature-time curve approached a position asymptotic to the latter variable it was difficult to obtain precise values indicating rate of fall of temperature.

The addition of an intermittent mechanical make and break of the circuit superimposed on the dilatometric temperature control radically assisted in smoothing out the curves. To introduce this intermittent control feature the cross member containing the upper half of the platinum contact *D* was pivoted about the point *F*. The other end of this lever rested on a rotating shaft *G* machined in the form of a hexagon. This shaft was attached to a clock motor and thus, rotating once every minute, produced a make and break in the circuit six times every minute. Thus the ratio of the "make" time to the "break" time became a substantially continuous function of the length of the rod and "hunting" was thus minimized.

The details of this mechanism are shown in Fig. 1, while a clear picture of the control apparatus is provided in Fig. 3.

The effects of the use of the modified control are given in Fig. 2, curves *C* and *D*, the temperature of experimentation being about 400 C.

A view of the complete test apparatus is shown in Fig. 4. The furnace to the right represents the first attempt and that to the left the last attempt to build a vertical furnace free from any marked temperature change over a 20-in. length. The first furnace was packed with monohydrated bauxite—a material decidedly superior to sil-o-cel in its heat-insulating power. While this was thermally very efficient, slight irregularities in packing rendered it extremely difficult to obtain temperature uniformity. A wider furnace using inferior insulating material (powdered alumina) seemed to give better results.

## MATERIAL USED IN THE TEST

The data in the present paper are restricted to those obtained in testing pure iron below 900 C. According to Mr. Wynne of the Chemical Division of the Westinghouse Research Laboratories, the chemical composition of the iron was as follows:

| | | | |
|---|---|---|---|
| Iron, per cent | 99.967 | Sulfur, per cent | 0.008 |
| Nickel, per cent | ...... | Silicon, per cent | 0.004 |
| Cobalt, per cent | ...... | Manganese, per cent | 0.002 |
| Copper, per cent | trace | Aluminum, per cent | ...... |
| Carbon, per cent | 0.001 | Oxygen, per cent | 0.016 |
| Phosphorus, per cent | 0.002 | | |

The specimens were prepared in the form of $\frac{3}{16}$-in. rods of uniform section by swaging and were thoroughly annealed before the test. The variations along the diameter of any test specimens were less than 0.001 in.

## FACTORS INFLUENCING TEST DATA

In the present type of creep test, there are certain factors which may influence the interpretation of the creep data obtained but which play no part in a consideration of the conventional type of creep test. Chief among these are the coefficient of expansion of the test rod and changes in elastic modulus. Temperature gradients are of importance in both forms of test. Space permits only brief reference to these factors.

In order to conduct a temperature-time creep test the material under test must have a positive coefficient of expansion with increase of temperature; otherwise the presence of creep leads to an elongation of the test rod, thus reducing the temperature, and this fall in temperature further increases the test rod length due to the negative coefficient of expansion. Once the contacts are broken, the temperature falls, therefore, continuously to that corresponding to the equilibrium conditions for the minimum applied voltage. If the coefficient is zero a similar condition holds. Further, if the coefficient of expansion is not constant in the range of temperature of experimentation, but say increases gradually with falling temperature, then the rate of fall in the temperature-duration curve will be decreased by an amount equivalent to the increase in the coefficient. A sharp break in the temperature-coefficient curve will be reflected in the slope of the temperature-time curve.

Since the temperature of test is constantly changing, the effect of change in elastic modulus must also receive attention. Data on the effect of temperature on elastic modulus by Koch and Dieterle[1] show that the modulus of iron falls from 20,600,000 to 15,000,000 lb. per sq. in. in raising the temperature from 0 to 600 C. More recent data from Lees Andrews and Shave[2] give the figures 20,300,000, 18,100,000 and 14,500,000 lb. per sq. in. for the temperatures 0, 300 and 600 C., respectively.

Consider the range between 300 and 600 C., where the temperature-modulus relationship approximates a linear form, and a few simple calculations will readily illustrate the relative importance of these two factors in their influence on the temperature-time curves which we present in this paper.

The change in elastic modulus due to 100 C. change in temperature will be found to be about 8 per cent.

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[1] K. R. Koch and R. Dieterle, "Die Elastizität einiger Metalle und Legierungen bis zu Temperaturen, die ihrem Schmelzpunkt naheliegen; B. Elastizitätsmodul," *Annalen der Physik*, Fourth Series, Vol. 68, No. 13, p. 441.

[2] Lees Andrews and Shave, "Modulus at Elevated Temperature," *Proceedings*, Physical Soc. (London), Vol. 36, p. 405; Vol. 37, p. 169.

It can be assumed that temperature changes affect expansion and modulus over the same length of test rod—about 40 in. Creep is restricted essentially to about a 20-in. length (Figs. 5 to 7).

Change in length due to change in elastic extension for 100 C. over 40 in. will be about 0.0013 in. for a load of 5000 lb. per sq. in. Length change for 100 C. due to temperature coefficient will be 0.06 in. (assuming a coefficient of 0.000015). Since both the thermal change and the modulus change function to alter the test rod length in the same direction, the total contraction due to 100 C. fall in temperature over a 40-in. length will equal the summation of 0.0013 and 0.06—a total of 0.0613. The percentage change due to change in modulus is thus only about 2 per cent of the total for the 5000 lb. per sq. in. load and proportionally less or greater than this for smaller or greater loads.

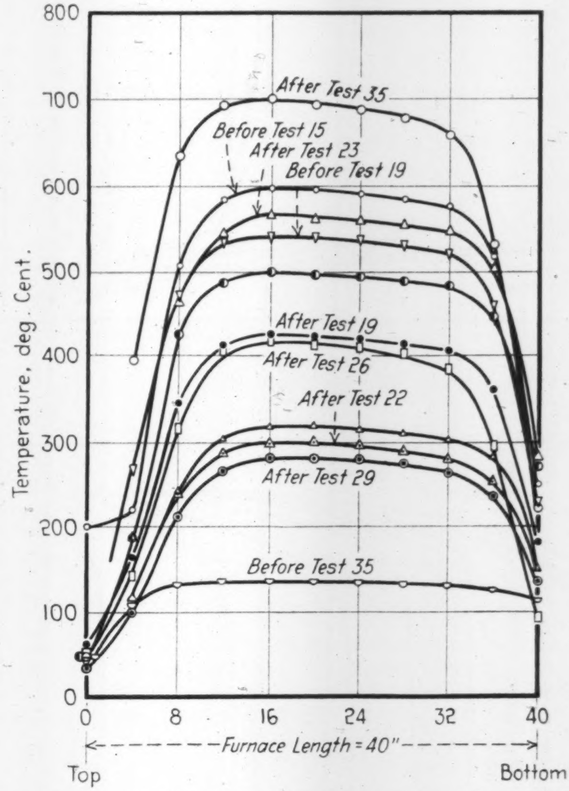

FIG. 5.—Temperature Gradients in Furnace No. 1.

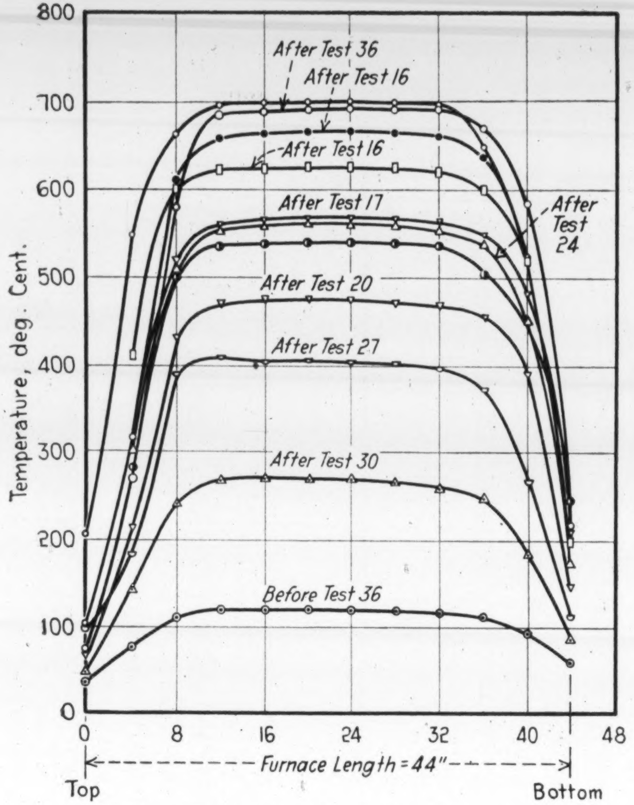

FIG. 6.—Temperature Gradients in Furnace No. 2.

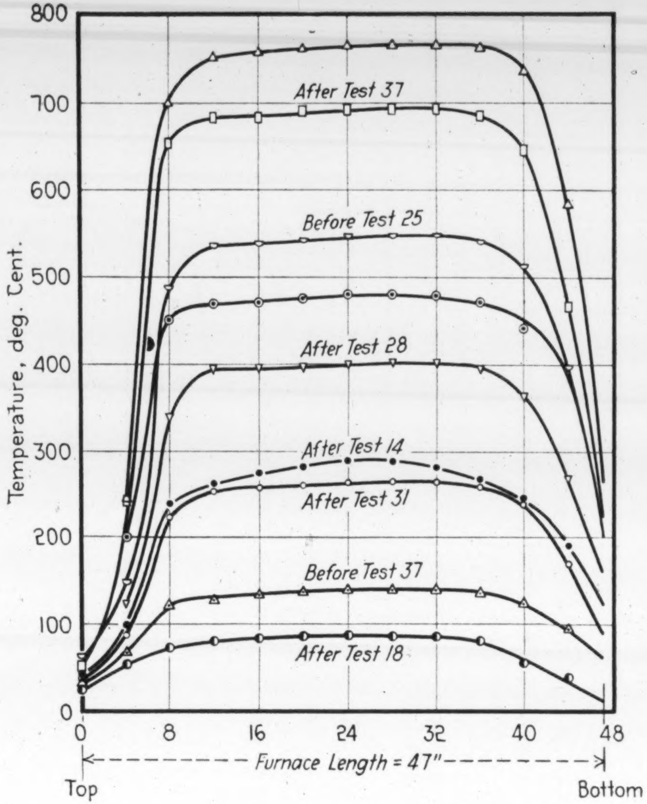

FIG. 7.—Temperature Gradients in Furnace No. 3.

It will be observed that the creep is considered to have taken place over only 20 in., although the furnaces measured about 40 in. in length. It was found practicable to maintain "constant temperature" only over about 20 in. Beyond these limits the temperature gradient was steep (see Figs. 5, 6 and 7). Since the laws connecting rate of creep and temperature for any given metal are not known, it is impossible to calculate the exact effect of these end gradients on the results obtained in test, but calculations on a first approximation indicate an error not greater than 10 to 15 per cent, part of which can be eliminated by corrections based on the known temperature distribution along the test specimen. Furthermore, a constant temperature over 20 in. in the strict sense was not obtained, although in several instances the variation was less than 10 C.

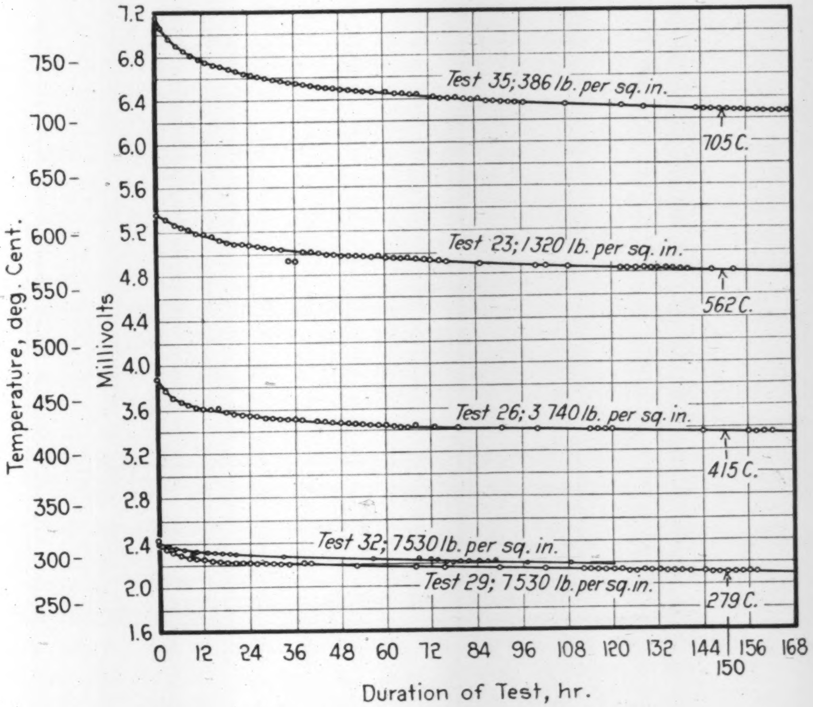

FIG. 8.—Creep Tests on Pure Iron in Furnace No. 1.

### RESULTS OF TESTS ON PURE IRON

The annealed swaged rod of the pure metal, about 54 in. long and $\frac{3}{16}$ in. in diameter, was screwed into the supporting block of the furnace and the predetermined load was applied, by use of the universal link, to the base of the bar. A platinum thermocouple was pushed up into the center of the furnace and a small stream (about $\frac{1}{3}$ cu. ft. per hr.) of hydrogen gas started. Thermocouple connections permitted temperature changes of 0.1 C. to be readily detected.

A suitable voltage was then imposed on the furnace windings, and as the temperature rose the screw contact fastened on the test rod (*A* in Fig. 1, see also Fig. 3) was screwed down to meet the lever holding the lower point of the pair of platinum contacts. This maintained the furnace at constant temperature unless the test rod began to exhibit perceptible creep. If creep did not lead to a few degrees fall in temperature within an hour or so, the screw contact was raised until the temperature had risen some 30 C., when the control was started again. If necessary this was repeated until a temperature had been reached where it was clear that the test rod was exhibiting definite creep under the initially applied load. A temperature record was then made every 2 hr. throughout the entire course of the test.

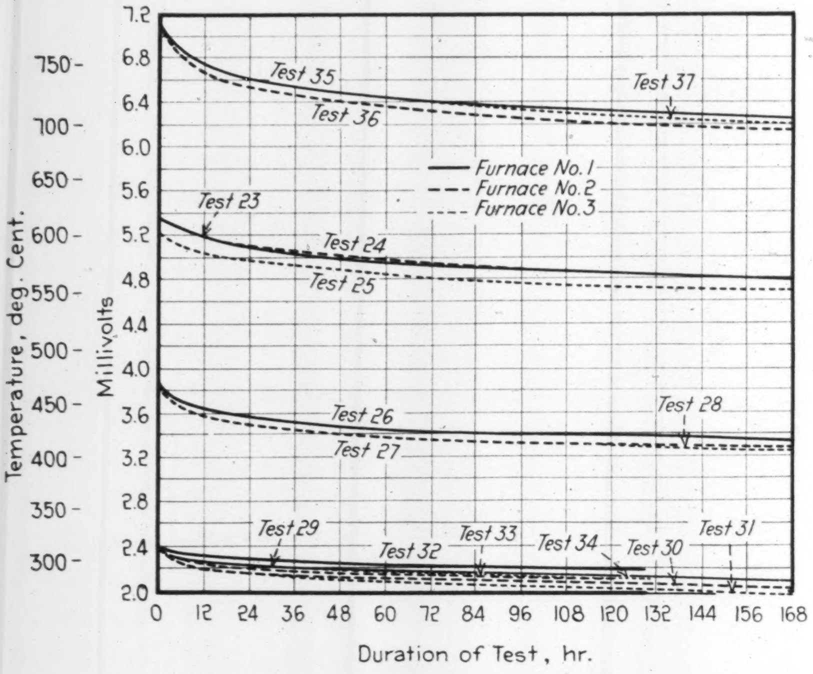

FIG. 9.—Summary of Creep Tests on Pure Iron.

It should be mentioned that the power-temperature relationships of all the furnaces was obtained before their application to creep testing. As soon as the suitable test temperature for the given metal and load had been determined as above, it was a simple matter to adjust the maximum and minimum voltages in accordance with the observations discussed in consideration of Fig. 2.

The temperature-time data obtained on pure iron have been plotted in Figs. 8 and 9. One test rod was placed in each of the three furnaces and loaded to 1320 lb. per sq. in. The tests were run concurrently for about one week. The load was then removed and the test rod annealed over night at 800 C.

The same rods were then loaded 3740 lb. per sq. in. at room temperature and the test repeated as outlined above for a similar period. A further anneal was followed by a test conducted in the same manner with a 7530 lb. per sq. in. load. All three bars were again annealed and retested with the 7530 lb. per sq. in. load in order to obtain data on the reproducibility of the test under identical conditions. Again the rods were annealed over night, cooled to room temperature, and loaded to 385 lb. per sq. in.

The actually observed data have been reproduced in these curves in order to give a true picture of the behavior of the test rod under constant load, and to indicate the "scatter" obtained. All curves on pure iron obtained in the three furnaces have been combined in Fig. 9, and it is instructive to compare this diagram with the relevant data on temperature gradients given in Figs. 5 to 7.

First it may be stated that the various furnaces provide results showing remarkably good agreement and that the limited differences are in the direction which would be anticipated. These differences, however, are in some instances less than would be expected. Thus consider tests 35, 36 and 37. Test 35 (Fig. 5) shows a marked gradient with a temperature difference of more than 30 C. over the 20-in. length. In test 36 (Fig. 6) there is no more than 5 C. variation while in test 37 (Fig. 7) a difference of nearly 15 C. may be noted. In the temperature-duration curves the maximum temperature was always recorded.

Since test 35 has a maximum temperature it is to be expected that perceptible creep would be limited to a short length of test rod. In test 36 perceptible creep should occur along a length of 20 in. Figure 9 illustrates that such is the case, although the temperature interval between the curves is small—20 C. However, on test work the temperature gradient conditions in furnace No. 1 during test 35 are regarded as wholly unsatisfactory, but it should be recalled that we are at this point examining only the test method and hence considering the magnitude of effect due to variations in test conditions.

A similar analysis can be focused on the remaining curves.

## COMMENTS ON THE TEST

The data we have presented have been confined to those necessary to indicate the functioning of the apparatus we have set up. In a later paper it is hoped to provide comparative data on the testing of other elementary metals and to discuss certain metallurgical investigations in which data obtained by this method provided a basis for derived criteria of the relative merits of the materials investigated.

It is considered that the addition of the intermittent mechanical make and break of the circuit superimposed on the dilatometric temperature control has resulted in the elimination of marked temperature fluctuations. One purpose of Fig. 8 is to illustrate exactly the quantity and nature of the data from which all the curves are drawn.

It cannot be too strongly emphasized that the importance of a test method largely depends on reproducibility of test data. Theoretically we can be assured that if the test herein described is employed to test exactly similar rods of a metal in furnaces exactly comparable with respect to temperature gradients, we shall obtain strictly comparable data. Whether it may be possible to procure reasonably similar test rods, the preparation of similar test furnaces is a matter of some speculation.

Thus in our test we aimed at producing a constant temperature over a 20-in. length with a steep gradient toward each end of the furnace. Practically this constancy of temperature is extremely difficult of attainment and the question naturally arises as to what temperature variance may be permitted in this "uniform temperature zone" without material modification in the test data obtained.

The general conception on the relationship between temperature and creep for any given load is that the rate of creep falls off very rapidly with small decrements of temperature. Thus it would be expected that most of the creep occuring in our test would be confined to any maximum temperature zone, and that a uniform temperature over 20 in. would exhibit markedly more creep and hence temperature fall than where such uniformity did not exist.

The purpose of Fig. 9 is to present data which were obtained in three different test furnaces exhibiting temperature gradients depicted in Figs. 5, 6 and 7, in order that some conception may be formed as to the effect of deviation from uniformity of temperature over the 20-in. length on the ultimate temperature-time curves resulting.

Having obtained satisfactory thermal and mechanical conditions, we have found that it is important to standardize test specimens along with the routine of initiating the test. These features will be discussed in a subsequent paper where attention will be drawn to the phenomenal effects on resistance to creep of minute amounts of strain hardening.

*Acknowledgment.*—The authors are indebted to the Westinghouse Electric and Manufacturing Co. for permission to publish the data obtained for this paper. They are pleased to acknowledge their appreciation of helpful criticism by Mr. P. H. Brace and of the supply of samples of pure iron by Mr. A. A. Frey.

# DISCUSSION

MR. G. M. EATON[1] (*presented in written form*).—Will the authors please outline their reasons for selecting the Rohn type of test instead of the Barr and Bardgett type? In selecting the type of test for our own needs, we narrowed the field down to these two. Our final selection of the Barr and Bardgett type was influenced largely by the prompt response to a change of stress as compared with the slower arrival at a state of balance when the temperature is changed. When brevity of test duration is the reason for adopting either type of test, some sound reason must exist for selecting the test requiring more time for stabilization after each control operation.

We see in Fig. 5 that as the furnace temperature increases, the length, over which the distribution is fairly constant, decreases. Compared with this, the uniformity of stress distribution is practically exact at any of the stress increments involved in the constant temperature test.

And finally, with the constant temperature test the various alloys undergoing comparison tests are tested at specific temperature steps, and at any one step the temperature distribution is closely similar regardless of the alloy being tested.

Fairness, however, demands the admission that we have had a great deal of difficulty in producing three weigh bars which are close to equality and which will remain constant.

MR. C. R. AUSTIN.[2]—The reason for the selection of the Rohn type of test instead of that by Barr and Bardgett was primarily due to the fact that we were acquainted with the former and not with the latter. I cannot discuss the relative merits of the two methods because I am not familiar with the varying load test.

However, it does appear that in the Rohn test we have a very simple means of differentiating between alloys. The test appears to present a ready means of sifting alloys and showing, in work on the development of new alloys, which materials are worthy of further test. Furthermore, we immediately collect data, over a continuous temperature range, which has definite metallurgical significance.

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[1] Director of Research, Spang, Chalfant and Co., Inc., Ambridge, Pa.

[2] Section Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

THE CHAIRMAN (*Mr. H. W. Gillett*)[1].—I should like to have further comments on the effect of a very small amount of deformation.

MR. AUSTIN.—The effect to which Mr. Gillett has reference is to be the subject of another paper so that I will make only passing reference. On account of the nature of the Rohn test, the profound effect of minute amounts of deformation on resistance to further creep is readily demonstrated. A specimen rod is heated to some predetermined temperature where creep takes place as soon as the load is applied. After a time the rate of creep falls to a very small value so the temperature of test is raised some fifty or sixty degrees. A similar creep curve is obtained with the temperature-time curve exhibiting a tendency to assume constant temperature after several hours.

Again the temperature may be raised some fifty degrees with similar result, but the "constant temperature" is considerably higher after each section of the test. This means that the temperature at which the rod can carry the load is markedly raised by the small amount of deformation induced by the successive stages of creep.

MR. GILLETT.—What amount of deformation are you talking about there? What percentage of deformation?

MR. AUSTIN.—I do not recall what the calculated amount of deformation happened to be, but it was something less than 0.001 per cent. The profound effect of these minute amounts of deformation will be discussed in a future paper.

MR. C. L. CLARK.[2]—What is the smallest length change on which the relay will operate; that will control the maximum sensitivity, will it not?

MR. AUSTIN.—Not altogether, since we have a time period over which the factors, which give us rate of creep, operate. These are the slope of the line and the coefficient of expansion. Assuming that a very definite and finite amount of change in length is necessary to operate the relay, this will result in a certain scatter of the data from which a mean curve is drawn. From this mean curve we obtain the slope which provides us with a temperature fall rate (such as 1 C. per 10 hr. time of test) which in turn affords information for determining rate of creep.

However, it is to be remembered that we have an intermittent mechanical make and break of the circuit superimposed on the dilatometric temperature control which eliminates the consideration brought out by Mr. Clark. We now have a condition where the clock motor supplies the motion for the actual make and break, while the extension of the rod merely determines the ratio of make time to break time. Thus, any minute extension of the rod causes a correspondingly small change in this ratio and hence in temperature of test.

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[1] Director, Battelle Memorial Inst., Columbus, Ohio.

[2] Research Engineer, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

From this it is apparent that there is no limiting sensitivity needed to effect make and break of contact.

MR. GILLETT.—Have you gone far enough to tell what the test is good for? In other words, does it give real indications?

MR. AUSTIN.—The present paper does not attempt to demonstrate the importance of the test in the evaluation of high-temperature properties of metals. It constitutes an effort to outline a test method alone, with the presentation of limited data on one metal—pure iron.

As regards the utility of the test, a simple example will be taken to illustrate the point raised. Assume we have four metals—iron, nickel, cobalt, and silver. A constant load is put on similar specimens of each metal and the temperature raised to some predetermined point above which creep is expected to occur. The specimen will elongate and the temperature fall in a ratio inverse to the resistance to creep of the metal, until we arrive at what may be termed the load-carrying capacity of the test bar.

We shall find that for some loads, nickel and cobalt approximate to a constant temperature of test at say 600 C. Iron will probably fall to 400 C. and silver to perhaps 150 C. Thus, the first approximation suggests these metals to be equally strong at the above-recorded temperatures. The superiority of nickel and cobalt as regards resistance to deformation over iron or silver at elevated temperatures is thus readily apparent. There are not sufficient data available at the moment to permit discussion of commercial alloys.

MR. H. J. GOUGH[1].—Although I understand that the modified Rohn test is not intended to replace the long-time creep test, there is one aspect of the test on which I should value the authors' views. I take it that the results obtained by this "short-time" test are ultimately required for information concerning the behavior of the same materials used at a constant temperature and load; in other words, under conditions employed in the normal "creep" test. Now, in the authors' test (Figs. 8 and 9) the temperature is dropping, at a decreasing rate, throughout the test. The result obtained from the test is interpreted, I understand, solely in relation to the final temperature. I wish to raise the question as to whether the creep behavior at this stage is not affected by the previous temperature history. If so affected, then the result obtained may not be directly comparable with the result of a test on the same material carried out at a uniform temperature throughout.

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[1] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

I mention this point because creep behavior at elevated temperatures appears to be so complicated, being affected by factors which are not of great importance in affecting the results of mechanical tests made at air temperatures. For example, at the National Physical Laboratory we have tested batches of steel from the same or precisely similar casts not distinguishable by chemical analysis or metallurgical examination. Yet their creep behaviors differed appreciably. It has emerged clearly that the creep behavior of a steel is a function not only of heat treatment and composition, but that the mechanical work performed during manufacture also affects the ultimate creep resistance. A final treatment such as normalizing does not produce a state of uniformity. I have had several experiences of different samples of one metal, which having been given a constant normalizing treatment after previous differing histories of cold working, have exhibited quite different physical properties. For these and other reasons, I do not feel convinced that the variation of temperature applied in the authors' test may not affect the result obtained and I should like to have their views on this point.

MR. AUSTIN.—We know that these heat treatments can profoundly affect materials, but it is also true in conventional creep tests that when the test is completed the material is certainly not the same as that on which the test was commenced. The important point brought out by Mr. Gough refers to structural changes due to change in temperature of test, but in the present test the temperature changes are limited to small decrements.

It is true of all creep testing or any test at elevated temperatures that we should have some metallurgical knowledge of the material on test, and unless we are passing from one structural field to another, it is difficult to accept any suggestion that moderate temperature changes during test are as important as the time factor which is inherent in any creep test.

MR. GILLETT.—It all depends on how you are going to define structural changes. If you are talking about carbide precipitation, a little structural change may throw you off very widely.

MR. AUSTIN.—The answer to Mr. Gillett lies in a reiteration of my previous remarks. The precipitation occurring in 18 per cent chromium, 8 per cent nickel alloy takes place in any type of creep test if conducted within the precipitation temperature range.

MR. GOUGH.—I do not consider that Mr. Austin has covered adequately the point I raised. I am concerned with the possible effect of the variation of the test temperature during the test on the behavior of the material at the end of the test. Supposing the initial and final temperatures are $T_i$ and $T_f$; the question is, will the behavior of the specimen when $T_f$ is reached be the same as if the test temperature had been maintained at $T_f$ throughout? I venture to suggest that, if this point has not yet received attention by the authors, it is worthy of investigation; until this is done, the value of the test will be open to doubt.

MR. GILLETT.—Will not yield strength tests carried out on a standard specimen in short-time high-temperature, or somewhat more extended tests give about the same information you get here? It would appear that it at least gives you an exploratory test to separate the sheep from the goats among the alloys. I was wondering whether the expensive equipment and the difficulty of getting suitable specimens from many of the alloys have compensating advantages.

MR. AUSTIN.—The method is particularly suitable for material that can be rolled and swaged. The specimen is long and thin for a cast alloy. In this test we do approximate to real creep conditions and measurements of flow due to creep. There appear to be no data available to permit any definite statement regarding the probably closer correlation between data obtained by the Rohn test and the creep test as opposed to those obtained by the short-time tension and the creep test.

MR. GOUGH.—As the relative merits of short and lengthy tests have been referred to, I would sound one word of warning, arising from our experience. The rate of creep is *not* the only factor of industrial importance. Cases are on record where failure by intercrystalline cracking has occurred after lengthy exposure times; this undesirable and highly dangerous characteristic may be entirely overlooked in a test of short duration.

MR. AUSTIN.—This is not a simple problem to discuss on account of the many factors involved. Intergranular cracking is well known and its study requires long-time testing. It is true that our test is short when considering intergranular oxidation failure which may take 40 days of test to be revealed. On the other hand, the ordinary creep test becomes "short" or inadequate when a period of say 40 weeks is required to reveal phenomena of the type mentioned.

It is considered that factors of this type are beyond the scope of the present suggestion for application of the test and in no way invalidate the possible useful application of the test as a means of "sorting alloys."

MR. E. E. THUM[1] (*by letter*).—It seems to me that much of the criticism directed against Messrs. Austin and Gier's paper misses the point, with the demand for a relationship between the Rohn test and the creep limit, whereas the authors have definitely stated that they are using the Rohn test merely as a rapid indicator of the metal's probable behavior under temperature and load, and not in an effort to evaluate numerically the relation between temperature, volume, and stress. As a method of getting quickly an approximate value, the Rohn test has the pitfalls of many another accelerated test, but that does not mean that the results of carefully controlled experiments may not be used in a rapid survey, especially when interpreted in the light of similar experiments which have been correlated with data from service.

An impertinent question may also be asked—"What is there sacred about the methods of creep testing adopted by this or that laboratory, that all must conform?" Even though most experimenters favor the plan of measuring small extensions of metal under constant temperature and constant load, such a plan does not inherently give any better idea of what to expect of a given metal than the plan of measuring small decreases in temperature necessary to keep it at a constant length under a constant load. Those who believe that the former plan is the more scientific should remember that two excellent laboratories, both using it, have reached opposite conclusions as to the relative advantages of 5 per cent chromium steel and plain carbon steel tubing for oil cracking still service (over 1000 F., 540 C.).

It is no proper criticism of Messrs. Austin and Gier's apparatus to say that the metal at the end of the test is in a different condition than it is at the beginning. That is also true of testing at a constant elevated temperature, even more so. It would appear that the metal is changed less by starting at a temperature just a little above where the metal is plastic and then reducing the temperature automatically to check this incipient plastic flow and achieving stability in that way, than by starting at a temperature in the same region and permitting the metal to flow plastically for a long time, until it strengthens itself against further elongation, and thus achieving stability by "cold work" (if such a term is applicable at 1000 F., 540 C.).

Either plan ought to give an indication of what may be expected in long service at load and temperatures. An *indication* is all we can hope to get anyway, and the results are of value to operators of high-temperature equipment only as indicators, to be checked against experience. Metallurgists and researchers must always remember that an operator cannot criticize a test because the metal changes during the test, for the metal in an oil cracking tube, for instance, after four years service, is not the same metal (structurally) as went into the still originally.

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[1] Editor, *Metal Progress*, American Society for Steel Treating, Cleveland, Ohio.

Whether the Rohn method or the more conventional method is the more easily controlled, gives more readily reproducible results, or gives a better idea of what happens at the "creep limit" are matters which might be discussed to advantage.

MR. AUSTIN (*author's closure, by letter*).—The authors appreciate the point of view taken by Mr. Thum in his post-meeting contribution to the discussion, and feel that it epitomizes many of the arguments which they themselves would wish to put forward as rebuttal of anticipated criticism of this new form of test for evaluating the resistance to plastic deformation in metals at elevated temperatures.

With reference to the comments by Mr. Gough, with respect to the possible effect of temperature variation during test, data available at present indicate that where we are free from constitutional changes the temperature range has little effect on the final result. When we pass, however, through a temperature range of age hardening, for instance, the test range of temperature can have a very marked effect on the end point.

The solution of such problems applies equally to conventional constant-temperature creep tests. Before creep testing is undertaken on any alloy it is manifestly important that some knowledge be gained of its constitution, and that an attempt be made to "stabilize" the alloy before testing. These data should provide information defining the permissible temperature range for the time-temperature type of creep test.

# THE TORSION IMPACT TEST

By G. V. Luerssen[1] and O. V. Greene[1]

## Synopsis

This paper points out the need for a reliable method of determining impact resistance on very hard materials such as hardened tool steel. It reviews briefly the work of previous investigators in this field, and comments on the inadequacy of present impact methods when applied to these materials.

A machine and method especially adapted to hard materials, in which the specimen is fractured torsionally under impact, are described in detail. Calibration of the machine, design of the test specimen, effect of velocity, and reproducibility of results are then discussed, and comparisons are drawn with current methods of test on softer materials.

Test data are shown on three hardened tool steels drawn over a range of hardness. These are discussed briefly, and general conclusions are drawn on the practical value of such data, and the utility of the machine as a control and research instrument.

## Introduction

There are many purposes for which extremely hard materials are used, which require in addition to hardness, a good degree of impact or shock resistance. Instances may be found in many of the more common tools such as taps, drills, blanking dies, etc., and in hardened parts used under impact as in the case of rock-drill equipment. It is more or less universally assumed that these extremely hard materials cannot be expected to possess also a high resistance to shock, and yet we know from experience that within a somewhat restricted range they frequently do differ considerably in shock value even for equivalent hardness values. We know further that such variations may be of sufficient magnitude to govern the success or failure of the tool or part in actual service.

Most of our knowledge of the shock values of these materials, however, has been of a qualitative character gleaned from observation of steel tools and hardened parts in service. It is obvious, therefore, that there must be a vast field in the quantitative measurement and control of impact resistance in hard materials which is still practically untouched. It is the purpose of this paper to describe an impact machine which is being used successfully in exploring this field, supplementing this with test results on several steels to illustrate its range of usefulness and to indicate its possibilities both as a research and as a control instrument.

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[1] **Metallurgical** Dept., Carpenter Steel Co., Reading, Pa.

## PREVIOUS WORK

A search of the previous literature reveals little published data upon the impact testing of hard materials. Grossmann,[1,3] Bain,[1] Barry[2,3] and Archer[3] show impact values on high-speed steel hardened and drawn at various temperatures. Both notched and unnotched bars appear to have been used in the various tests recorded. While results of the various investigators are not in entire agreement due possibly to the variance in methods used, they all indicate quite definitely a maximum at a drawing temperature of 900 F. (480 C.).

Margerum[4] has described a method of measuring the shock strength of hardened steel in which a test bar is broken transversely under impact, the maximum pressure exerted being measured by the indentation of a ball in a standard block. The results are expressed in pounds pressure on the specimen, and not in energy absorbed. Tests made on a 1.08 per cent carbon steel hardened and drawn at increasing temperatures show a gradual increase in strength up to a drawing temperature of 600 F. (315 C.), then a gradual decrease to the annealed condition.

Styri[5] shows Charpy impact values on a ball-bearing steel of approximately 1.00 per cent carbon and 1.50 per cent chromium, hardened from different temperatures, and drawn over a range of temperatures. For each hardening temperature the impact resistance increases uniformly with ascending drawing temperature.

Probably the most important recent contributions to our knowledge of the toughness of hardened steel are the three papers recently published by J. V. Emmons,[6,7,8] in which a static torsion method is employed, the result being expressed in terms of a calculated figure known as "coefficient of toughness." Mr. Emmons' first paper appeared when the authors were accumulating their first data upon the impact resistance of carbon tool steel, and they were quite gratified to find a marked similarity between their own curves in which impact resistance is plotted against drawing temperature and those of Mr. Emmons in which coefficient of toughness is plotted as ordinate.

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[1] M. A. Grossmann and E. C. Bain, "High Speed Steel," p. 140, John Wiley & Sons (1931).

[2] R. K. Barry, "Hardness and Toughness of High Speed Steel as Affected by Heat Treatment," *Transactions*, Am. Soc. Steel Treating, Vol. 10, p. 257 (1926).

[3] R. K. Barry, "Comparisons of Impact and Slow Bend Tests of High Speed Steel," *Transactions*, Am. Soc. Steel Treating, Vol. 12, p. 630 (1927).

[4] C. E. Margerum, "A Test for Shock Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 876 (1921).

[5] Haakon Styri, "Relation Between Magnetic Properties, Impact Strength and Hardness," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 94 (1931).

[6] J. V. Emmons, "Some Physical Properties of Hardened Tool Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 47 (1931).

[7] J. V. Emmons, "Some Physical Properties of High Speed Steel," *Transactions*, Am. Soc. Steel Treating, Vol. 19, p. 289 (1932).

[8] J. V. Emmons, "Some Molybdenum High Speed Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, p. 193 (1933).

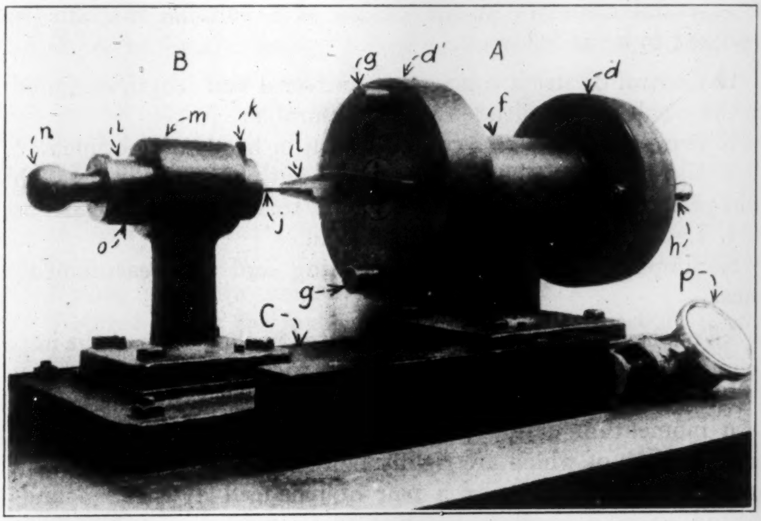

FIG. 1.—View of Machine with Specimen Set Up Ready for Test.

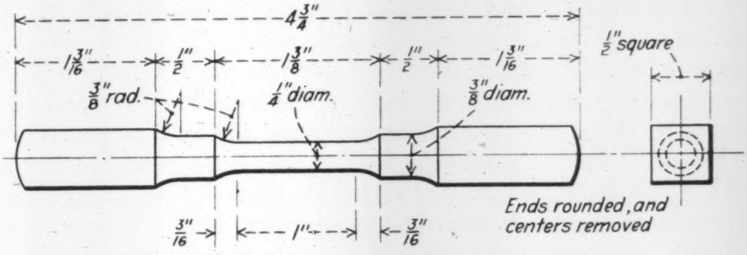

FIG. 2.—Details of Torsion Impact Specimen.

## DESCRIPTION OF MACHINE AND METHOD

In studying the previous attempts to secure impact values on hardened steels, it appeared to the authors that the principal difficulties lay in the use of a machine and a method primarily designed for materials of higher ductility. In general, these methods employ a notched transverse bar, an unnotched transverse bar, or an unnotched tension bar. Fundamentally, of course, all of these employ suddenly applied tensile stress, concentrated in varying degrees depending upon the design of the specimen, notch, etc. In all cases, however, the stress concentration is too great to allow the accurate and ready measurement of impact values on low ductility material, and the authors consequently resorted to the torsion principle which provides a means of sufficient stress distribution to facilitate measurement. The essential elements in the design of a suitable machine were considered to be as follows:

1. Control of stress concentration over a test length designed to give the results a magnitude easily measurable.
2. Prevention of stress concentration in heads of specimen.
3. Use of a specimen having a test length and section area which would permit uniform and successful heat treatment in any steel used.
4. Low cost in specimen preparation.
5. Simplicity and facility in testing and in measurement of values.

All of these elements were incorporated in the design of the impact machine and test specimen illustrated in Figs. 1 and 2.

Referring to Fig. 1, the machine consists of two major parts, first a momentum unit, *A*, and second a head, *B*, for holding the specimen, both of which are rigidly bolted to the bed plate, *C*. The momentum unit consists of a pair of balanced flywheels *d* and *d′*, mounted upon a shaft which is free to rotate in a ball bearing mounted in the housing *f*. The flywheel *d′* carries on its outer face two symmetrical striking bosses *g*. The flywheels may be rotated by means of the counter-balanced handle *h*.

The head, *B*, is made up of a holder sleeve, *i*, having a square hole in one end, into which the head of the specimen *j* is clamped by means of set screw *k*. To the opposite end of the specimen is attached the crossarm *l*, having a square hole into which the head of the specimen is snugly fitted and secured with a set screw. The holder sleeve *i* can be slid axially in the housing *m* by means of the knob *n*, and is prevented from rotating by the key *o*. The extent of the axial movement of the sleeve and specimen is so limited, that when the slide is pulled entirely back, the crossarm *l* will clear the bosses *g*, and when it is pushed forward to its fullest extent it will almost touch the face of the flywheel *d′*.

The actual operation of making the test is essentially as follows: The test specimen is clamped in the holder sleeve *i*, as already described, with the crossarm *l* properly attached. The knob *n* is pulled entirely back so that the cross arm *l* entirely clears the bosses *g*. The flywheels

$d$ and $d'$ are then rotated by means of the handle $h$ (or when very high speeds are required by a friction wheel driven by a portable motor on the periphery of $d$) to a predetermined speed measured by a portable tachometer driven on the periphery of $d$. The knob $n$ is then pushed sharply inward, bringing the crossarm into engagement with the bosses on the rotating wheel, and the specimen is thus broken under torsion impact. The residual speed of the wheel is then read, and since the kinetic energy corresponding to various wheel speeds has been previously calculated from the moments of inertia of the various moving parts, the energy absorbed in foot-pounds in breaking the specimen can be readily obtained from a prepared table. From the above description it is evident that the actual operation of making the test is extremely simple. One operator only is required, and the entire operation of mounting and breaking the specimen and obtaining the result requires only about two minutes.

Details of the specimen are shown in Fig. 2.

Figure 1 shows the machine with the specimen set up ready for test and the portable tachometer, $p$. A guard made of thin sheet screwed to the flywheel $d'$, and extending beyond the striking bosses, prevents the crossarm from flying after the specimen is broken. A guard made of wire gauze is swung over the middle portion of the machine during testing to prevent the flying of fragments. For simplicity both of these guards have been omitted from the illustration.

*Calibration of Machine:*

The speed-energy relationship of a rotating body is expressed by the equation:

$$K = \frac{1}{2}\,\omega^2 \frac{W}{g}\,\rho^2 \qquad (1)$$

in which $K$ is kinetic energy, $\omega$ is radians per second, and $\frac{W}{g}\rho^2$ is the mass moment of inertia.

In the machine described above, the moments of inertia of the flywheels, striking bosses, handles and shaft were calculated separately and added, and from this sum the moment of inertia of the crossarm was subtracted. The total moment of inertia of all rotating masses in pounds-feet was found to be 0.093645.

Since $\frac{1}{2}\,\omega^2 = 0.0054831\,S^2$, in which $S$ is expressed in revolutions per minute, Eq. 1 becomes:

$$K = 0.000513664\,S^2 \qquad (2)$$

where $K$ is expressed in foot-pounds.

From Eq. 2, energy values were determined for increments of 20 r.p.m. indicated speed for the particular tachometer used. This corresponds to about 4.7 actual revolutions per minute. Typical speed-energy conversion values using a tachometer with a 2-in. diameter wheel are as follows:

| INDICATED PERIPHERAL SPEED, R.P.M. | ACTUAL SPEED, R.P.M. | KINETIC ENERGY, FT-LB. |
|---|---|---|
| 2020 | 477.0 | 116.8 |
| 2080 | 491.2 | 123.9 |
| 2140 | 505.4 | 131.2 |
| 2200 | 519.5 | 138.6 |

If desired, the moment of inertia of the rotor may also be determined experimentally. One possible method consists in unbalancing the rotor by a known weight, the gravity axis of which is a known distance from the axis of the rotor. By observing the period of the compound pendulum thus formed, the moment of the rotor may be calculated from the pendulum equation. A second method, and possibly a more accurate one, consists in removing the rotor from the machine and suspending it by a wire attached to its axis, thus producing a torsion pendulum, from which the moment of inertia can be derived from Hooke's law by comparison with a similar pendulum of known moment of inertia.

*Test Specimen:*

The test specimen shown in Fig. 2 has been brought to its present stage of development both by a systematic study of various sizes and shapes, and as a result of steady evolution over a long period of routine testing. The diameter of $\frac{1}{4}$ in. was adopted to insure a uniformly hardened test section in the shallowest hardening steels such as the plain carbons and the carbon vanadium types. A test length of 1 in. was selected as being sufficient to afford considerable energy absorption, yet not of such extreme length as to cause warpage in hardening. The effect of varying length upon absorbed energy on test specimens otherwise identical is as follows:

| | STEEL A | | STEEL B | |
|---|---|---|---|---|
| GAGE LENGTH, IN. | IMPACT, FT-LB. | ROCKWELL HARDNESS NUMBER, "C" SCALE | IMPACT, FT-LB. | ROCKWELL HARDNESS NUMBER, "C" SCALE |
| 1 | 8, 9 | 66.5 | 148, 155 | 63.2 |
| 2 | 11, 13 | 66.0 | 247, 247 | 63.1 |
| 3 | 15, 16 | 66.0 | 353 | 63.3 |

A $\frac{1}{2}$-in. square head was selected as giving sufficient rigidity to the ends without introducing undue quenching hazards due to disparity between section areas. It was found desirable to chamfer or fillet all sharp edges and corners to prevent breakage in the shoulders both in hardening and in testing. Heavy tool marks are carefully avoided in machining, particularly in the test section and fillets, and, after treating, the test section is polished free from scale and pits by hand, using No. 150 emery cloth. The final diameter of the test section is held to ±0.002 in. The test specimen is inexpensive to prepare and could probably be made even more simple by the use of round ends having one side milled flat to conform to a similar change in the design of the holder.

*Effect of Velocity:*

A careful study was made also of the effect of initial velocity of the wheel upon the values obtained, by testing a series of identical specimens using different initial energies. The following table shows such a series, indicating that the effect of variable initial speeds is negligible:

| INITIAL SPEED, R.P.M. | INITIAL ENERGY, FT-LB. | AVERAGE IMPACT, FT-LB. | AVERAGE ROCKWELL HARDNESS NUMBER, "C" SCALE |
|---|---|---|---|
| 391 | 77 | 34.4 | 63.6 |
| 472 | 111 | 33.7 | 63.7 |
| 543 | 147 | 36.1 | 63.7 |

NOTE.—Data based on eight tests at each speed.

These results have been checked on numerous occasions in the course of routine testing, and it is safe to say that the only restriction on initial speed is that it be of such magnitude that the residual energy in the flywheel after fracture of the specimen is at least approximately 25 per cent of the initial energy.

*Reproducibility:*

Refinements in test-specimen design described in the preceding paragraphs have resulted in very good reproducibility, as indicated in actual duplicate readings observed in the course of the work. To obtain additional mass data, 670 tests upon which duplicate specimens were broken were taken at random and the high and low readings totaled. The total of the high readings was 23,896 and of the low readings 22,485, giving an average divergence as calculated on the high readings of 5.9 per cent. Similar data obtained on 556 duplicate Izod tests made with the standard notched bars[1] upon medium and low-carbon alloy steels, showed an average divergence of 8 per cent. Comparison of these data (not given in detail here) shows that reproducibility of results by the torsion impact method on hardened tool steels compares very favorably with that of the Izod method on softer steels.

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[1] A specimen 0.3937 in. square with a notch having a radius of 0.010 in. at the bottom, an angle of 45 deg. between the sides, and a depth of 0.0787 in.

## TEST DATA

In order to illustrate the field of usefulness of the machine and to make some direct comparisons between the torsion impact method and the usual impact methods, characteristic test results upon three steels—namely, a plain carbon steel; a ball-bearing steel containing 1.03 per cent carbon and 1.50 per cent chromium; and a regular 18 per cent tungsten, 4 per cent chromium, 1 per cent vanadium high-speed steel—are presented in the following paragraphs:

*Plain Carbon Steel:*

Figure 3 shows notched Izod, unnotched Izod, torsion impact values, and Rockwell hardness readings on a plain carbon tool steel hardened and drawn at different temperatures up to 800 F. (425 C.). The steel selected was of the fine-grained tough-timbre type, having the following analysis:

| | | | |
|---|---|---|---|
| Carbon, per cent | 1.06 | Sulfur, per cent | 0.012 |
| Manganese, per cent | 0.20 | Chromium, per cent | 0.03 |
| Silicon, per cent | 0.16 | Nickel, per cent | 0.04 |
| Phosphorus, per cent | 0.010 | | |

Test specimens were cut from $\frac{1}{2}$-in. square bars, spheroidize annealed to a Brinell hardness of 160. All test specimens after machining were preheated to 800 F. (425 C.) then transferred to a lead pot, heated uniformly to 1450 F. (790 C.) and quenched in brine. Specimens were drawn for 1 hr. at the respective temperatures shown. The undrawn specimens were tested 24 hr. after quenching. Izod specimens were of standard design, the notches having been ground after treating and drawing. All tests were made in duplicate. It might be added that comparisons were also made upon Izod specimens having a notch with a radius of $\frac{1}{16}$ in. and a depth of 0.0805 in., treated after the notches were cut. These showed slightly higher values than did the sharp notch but are not reported since they do not represent standard conditions.

The difference in the impact curve obtained on the torsion machine and upon the Izod is quite striking, and particular attention is called to the peak occurring on the torsion curve at a drawing temperature of about 350 F. (175 C.), a peak which is entirely missing in the other types of test. This peak is quite similar to those shown by Emmons[1] for a steel containing 1.12 per cent carbon and 1.07 per cent chromium, and is quite characteristic of all the extremely hard steels thus far tested, its exact location and height depending upon a number of factors which will be touched upon later.

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[1] J. V. Emmons, "Some Physical Properties of Hardened Tool Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 47 (1931).

The character of the fracture obtained at different points on the curve is quite interesting, and is illustrated in Fig. 4 which shows the series of specimens used in compiling the curve shown in Fig. 3. It will be noted that from the as-quenched condition up to a drawing temperature of 275 F. (135 C.) the fractures are of the rough or shattered type. These test specimens literally explode during the test. From 325 to 350 F. (165 to 175 C.) the specimens first shear, and then shatter. On specimens drawn at 375 F. (190 C.) and higher, the fractures are distinctly of the shear type, much the same as fractures obtained on soft steels broken in static torsion. Steels in this range often take a considerable plastic set before fracture. Cases have been encountered in which the initial energy has not been sufficient to break the specimen, and in some such instances as much as 90 deg. twist has been noted, even with a Rockwell hardness, "C" scale, as high as 63.

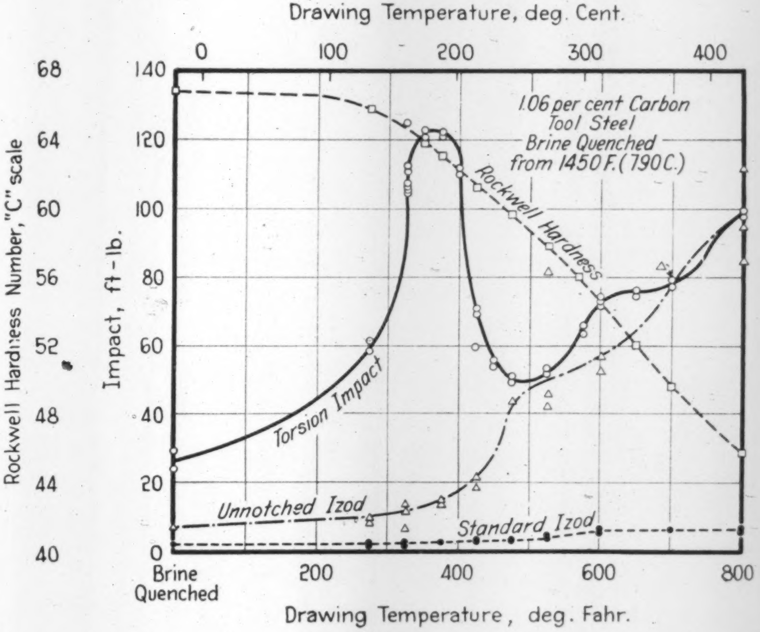
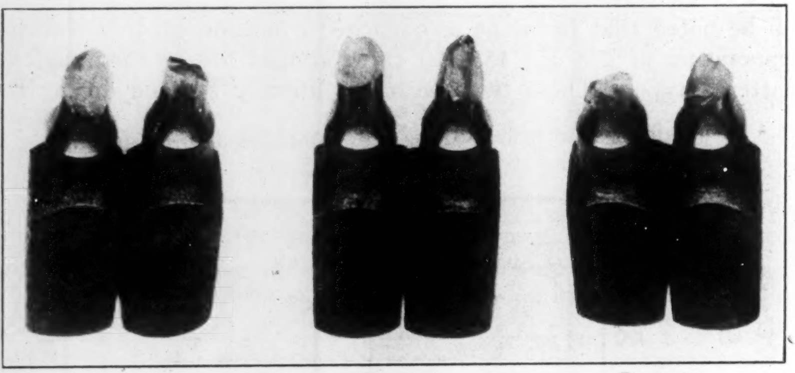


FIG. 3.—Relation Between Izod and Torsion Impact Tests on Plain Carbon Steel, Brine Quenched from 1450 F. (790 C.) and Drawn as Shown.

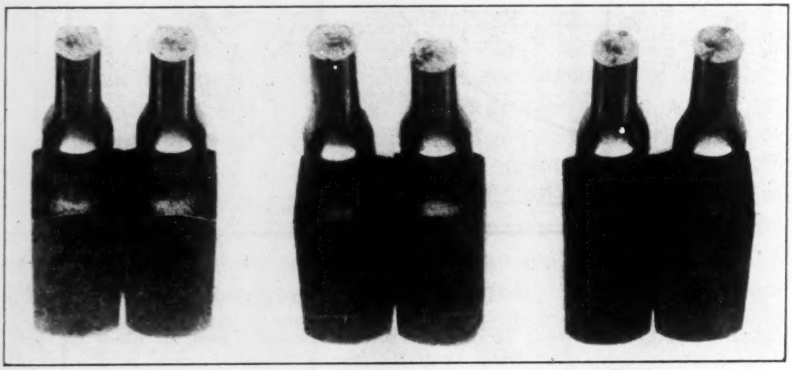
The rise of the curve from no draw up to about 325 F. (165 C.) is probably due partly to stress relief and partly to critical carbide dispersion in the martensite, the structures corresponding to the peak being uniformly martensitic. The structures in the falling portion of the curve from 375 to 475 F. (190 to 245 C.) are duplex, martensite and troostite, while those beyond 475 F. (245 C.) at which point the curve again begins to rise are troostitic. Quenching temperature has quite a marked effect upon the position of the peaks and valleys of this curve. In general the higher the quench, the higher will be the drawing temperature at which the various changes in the curve occur.



As Quenched

Drawn at
275 F.
(135 C.)

Drawn at
325 F.
(165 C.)



Drawn at
375 F.
(190 C.)

Drawn at
425 F.
(220 C.)

Drawn at
475 F.
(245 C.)

FIG. 4.—Fractures of Straight Carbon Torsion Impact Specimens Brine Quenched from 1450 F. (790 C.) and Drawn as Shown.

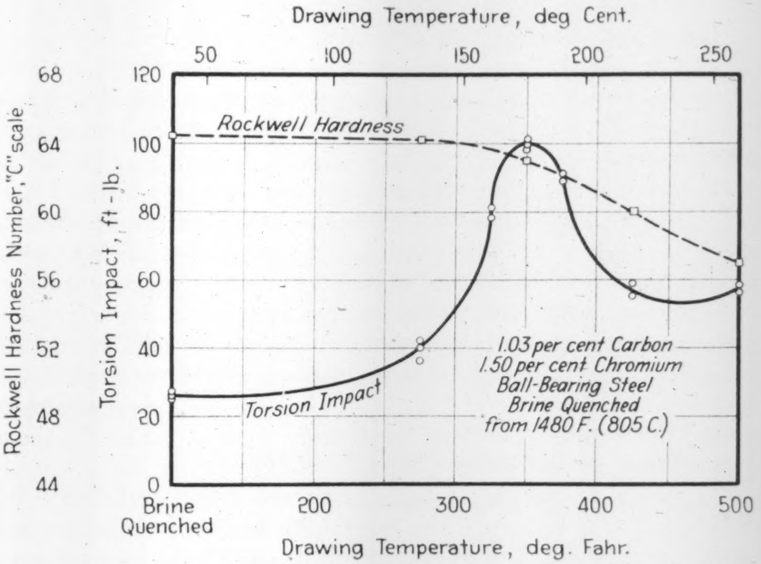


FIG. 5.—Torsion Impact Results on Chrome Ball-Bearing Steel, Brine Quenched from 1480 F. (805 C.) and Drawn as Shown.

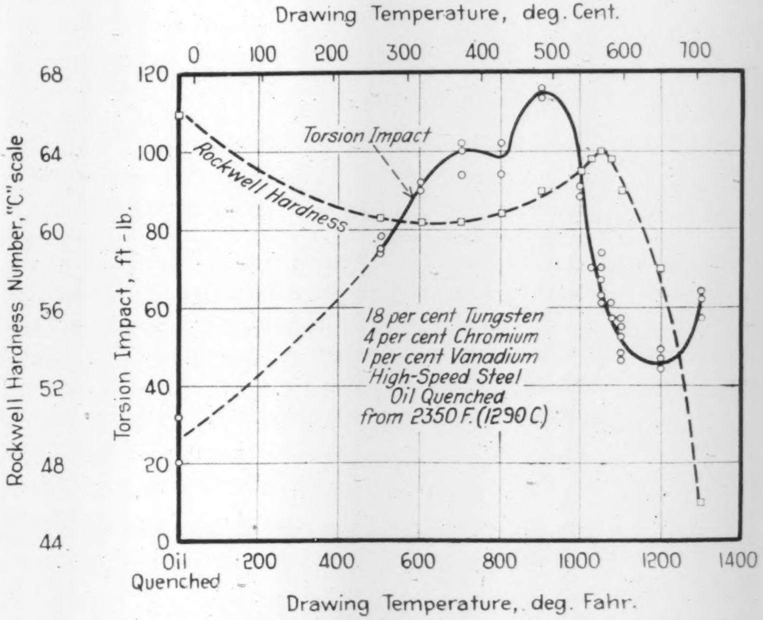


FIG. 6.—Torsion Impact Results on 18 per cent Tungsten, 4 per cent Chromium, 1 per cent Vanadium, High-Speed Steel Oil Treated from 2350 F. (1290 C.) and Drawn as Shown.

Due to the sensitive character of the test, many variables which have not previously been considered in quantitative testing of very hard steels but which nevertheless have been suspected as important in actual service, can be detected, and must therefore be taken into consideration if significant results are to be obtained. For instance, the character of the atmosphere in the hardening furnace has a distinct influence on impact resistance, and the specimens used in the present discussion were consequently quenched from the lead pot to eliminate this variable. Time of soaking at the quenching temperature, length of time at drawing temperature, and character of the initial structure all have been found to influence the results. In addition to all of these, the timbre characteristics or inherent hardenability of the steel itself has a very profound influence which is superimposed upon all the variables already mentioned. The scope of the present paper will not permit more than a passing notice of these variables affecting the shape of the impact curve, and it is hoped that a more detailed account of these very important factors will be presented sometime in the future. They are mentioned here to indicate the vast potential field of practical research which it is obviously possible to explore by this method.

*Chromium Ball-Bearing Steel:*

Figure 5 shows curves plotted from data determined on a ball-bearing steel of the following analysis:

| | | | |
|---|---|---|---|
| Carbon, per cent | 1.03 | Sulfur, per cent | 0.014 |
| Manganese, per cent | 0.36 | Chromium, per cent | 1.50 |
| Silicon, per cent | 0.23 | Nickel, per cent | 0.08 |
| Phosphorus, per cent | 0.023 | | |

Specimens were made from ½-in. square bars spheroidize annealed to a Brinell hardness of 170. They were hardened by preheating at 800 F. (425 C.), then transferring to a lead pot heated to 1480 F. (805 C.), held 20 minutes at temperature and quenched in 15-per-cent brine. They were subsequently drawn for 1 hr. at the temperatures indicated. The form of the curve is seen to be quite similar to that of the plain carbon steel.

*High-Speed Steel:*

Figure 6 shows curves plotted from data determined upon a high-speed steel of the following analysis:

| | | | |
|---|---|---|---|
| Carbon, per cent | 0.72 | Sulfur, per cent | 0.017 |
| Manganese, per cent | 0.22 | Chromium, per cent | 3.79 |
| Silicon, per cent | 0.22 | Tungsten, per cent | 18.77 |
| Phosphorus, per cent | 0.025 | Vanadium, per cent | 1.04 |

Specimens were machined from rolled bars $\frac{1}{2}$ in. square annealed to a Brinell hardness of 217. All specimens were hardened at one time from a gas-fired furnace so as to insure a constant atmosphere. Pieces were preheated to 1600 F. (870 C.), transferred to the high-temperature zone, heated rapidly to 2350 F. (1290 C.), and quenched in oil. They were then drawn for 1 hr. at the temperatures indicated.

Comparison of this curve with that determined by Emmons[1] for a high-speed steel similarly treated and those determined by Barry,[2] Grossmann[2] and Archer[2] will be found interesting. There appears to be a fair agreement on the peak at 900 F. (480 C.) and the valley at 1100 to 1200 F. (595 to 650 C.), although the authors' curve shows this to occur at a slightly lower drawing temperature than those of previous investigators.

## SUMMARY AND CONCLUSIONS

In view of the results presented it is felt that the previous methods of impact testing on hard materials have not always yielded results indicative of the true impact resistance of these materials, on account of variable stress concentration resulting from the character of the specimen and load application. It is believed that the torsion impact method described not only obviates these difficulties, thus providing a means for making comparisons of impact values, but also operates with an accuracy and ease commensurate with standardized methods used on softer materials.

The test data presented on three different types of steel indicate some very important and fundamental characteristics in hardened tool steel, a more complete knowledge of which should make possible a more intelligent study of tool problems in two ways.

1. Through the selection of a proper tool steel both from the standpoint of analysis, and timbre; and
2. Through the use of heat treatment methods designed to bring out the best properties of the steel selected.

It is therefore felt that the results presented will open a field of testing not hitherto explored in a quantitative way, and should be a means of assisting those interested not only in hardened tool steels, but in other hard materials as well, in more definitely laying out their requirements.

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[1] J. V. Emmons, "Some Physical Properties of High Speed Steel," *Transactions*, Am. Soc. Steel Treating, Vol. 19, p. 289 (1932).

[2] R. K. Barry, "Comparisons of Impact and Slow Bend Tests of High Speed Steel," *Transactions*, Am. Soc. Steel Treating, Vol. 12, p. 630 (1927).

# DISCUSSION

MR. HOWARD SCOTT[1] (*presented in written form*).—The torsional impact test, as well as Emmons' static torsion test, has revealed clearly a state of maximum torsional toughness in slightly tempered hardened tool steels. This is a fact of many implications which opens up new leads of metallurgical as well as mechanical significance. Little or no evidence of a tough condition in this tempering range is yielded by tests which cause failure on bending or tensile loading. Evidently there is a basic difference between the mechanism of failure under torsion and that under tension impact which is worthy of attention.

Although it is not safe to conclude that tempering to maximum torsional toughness gives unusual bending impact strength, one might expect it to do so under favorable conditions. A steel of lower carbon content tempered in the same temperature range provides a more favorable condition than that reported by the authors. The writer has made impact tests on a 0.4 per cent carbon steel as hardened in the form of standard Charpy specimens except for the notch which was ground and lapped to 0.079 in. deep with a radius of 0.062 in. Indications of that effect were found in the form of a rapid rise in impact strength on tempering just above 150 C. followed by a flattening of the curve.

The maximum in torsional impact strength at low tempering temperatures attracts attention from a metallurgical viewpoint also. The maximum is reached when or just before the transformation of residual austenite to martensite on tempering is completed. According to an explanation of this phenomenon advanced by the writer,[2] the fact that austenite transforms at this temperature indicates that restraining pressure on the austenite has relaxed. Consequently the microresidual stresses after tempering should be greatly reduced with advantage to ductility and impact strength.

Tempering at temperatures beyond the peak, the torsional impact strength drops abruptly. This is a phenomenon of exceptional interest, for the ductility measured either by static or impact torsion tests falls with falling hardness instead of rising. The same phenomenon was apparently observed first in connection with the aging of duralumin where it is rather prominent. In fact, it appears to be quite generally associated with age-hardening. For example, it is seen in the authors' high-speed steel curves, Fig. 6, after peak hardness, that is, secondary hardening is reached. The secondary hardening phenomenon in high-speed steel is unquestionably chiefly an age-hardening reaction.

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[1] Section Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Howard Scott, "Dimensional Changes Accompanying the Phenomena of Tempering and Aging Tool Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 9, p. 291 (1926).

From the preceding observations, it appears that age-hardening plays a prominent rôle in the reactions of steel tempering, the conventional peak hardness observed on tempering an alloy susceptible to age-hardening being obscured by the high initial hardness of quenched steel. The high initial hardness is undoubtedly due to some other cause and would decline sharply with tempering were the hardness trend not arrested by the mounting effects of age-hardening. Apparently, then, the observed hardness - tempering temperature curve is a composite of these two reactions on which is superimposed the transformation of residual austenite. As is usually the case, new test methods and information are required to throw in relief the complexity of familiar phenomena, such as tempering of steel.

MR. H. S. RAWDON.[1]—I should appreciate it if the authors would amplify their explanation of the reason for the peak in the curve showing the relation between torsional impact resistance and the tempering of the steel. If the changes in impact resistance were progressive, it would not be difficult to tie them in with the structural changes which are known to take place during the tempering of steel. The sharp rise in impact resistance followed by a sharp drop and this, in turn, by another rise, however, all occurring in a steel as the degree of tempering is being progressively increased do not fit in with what we know concerning the structural changes during tempering. The point is an important one and it would be helpful if the authors can throw any further light on this obscure point.

MR. HAAKON STYRI.[2]—I should like to ask how Mr. Greene determined that there was only pure martensite in the specimen after tempering it at 350 F. (175 C.). It is really very important and a very interesting point. It is of course possible to determine the quantity of martensite and austenite by X-ray, and it can also be determined by volume changes or dimensional changes. It is also of interest to know what type of martensite is present, and all these structures should be correlated with the obtained results. We are doing some work on the influence of tempering time and in a year or two may have something to report.

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[1] Chief, Division of Metallurgy, U. S. Bureau of Standards, Washington, D. C.

[2] Director of Research, S. K. F. Industries, Inc., Philadelphia, Pa.

MR. A. L. DAVIS.[1]—The sharply defined region of optimum drawing temperature brought to light by the work of Messrs. Luerssen and Greene arrests the attention of the user of tool steel. A similar narrow tempering range, of notably high energy-absorbing capacity, has been found by the makers of what is known as "gun shield plates" which must be of minimum possible thickness that will protect field gunners from rifle fire. In this case, insufficiently drawn plates will shatter like glass, then, with slightly higher drawing temperature, occurs the narrow range where success is found, and at only a little higher heat still the plate shears like cheese. The optimum plate, if overtaxed by too great weight or too high velocity of bullet, fails much as do the optimum tests of the authors, where some stretch, partial shearing, and eventual cracking characterize the testing to destruction.

Investigators of tool steel, who have previously relied largely upon Mr. J. V. Emmons' test for measuring toughness of hardened steels, are now given another method, which introduces the valuable feature of sudden load application, and thus constitutes a shock-resisting test, as well as a measure of strength and ability to withstand deformation before fracture in hardened steels.

MR. A. V. DE FOREST.[2]—I think this test has very great possibilities if it will illuminate the question of what happens to the internal stresses in hardened steel. We know that hardened steel before drawing may be softer than it will be after a small amount of drawing, and yet it is very much more brittle, shatters and flies apart, and, in general, acts as though filled with internal stress. Then, as the tempering proceeds, perhaps the internal stress is dissipated more rapidly than the material is softened, and perhaps this test is one of the very few, perhaps the only test we know of, which will enable us to study the dissipation of those internal stresses. It seems reasonable to suppose that there must be some mechanical test that can show how those stress conditions are dissipated, and a degree of toughness produced. Unfortunately, the notched-bar toughness, or the tensile impact toughness, does not show us at all how the internal stress is removed and the improvement that we would expect to find theoretically takes place.

MR. ARCHIBALD HURTGEN.[3]—I should like to emphasize the importance of temperature and time upon the toughness of a piece of steel. The impact tests certainly portray definite toughness characteristics of a piece of steel which may be affected by the temperature-time factor of the draw. It is possible to take several pieces of tool steel which have been properly quenched and drawn at say 400 F. (205 C.) for one hour, or drawn at 350 F. (175 C.) for an increased number of hours, thereby producing approximately the same hardness as a result of the two different drawing cycles, but the one having the longer draw at the lower temperature possessing considerably more toughness. For certain steel needles as used in the textile industry, where it was necessary for a needle to be very tough so as to permit it being bent through a considerable angle and at the same time be sufficiently hard so as to give good life, it was found necessary to prolong the drawing time at a low temperature for some twelve hours, whereas shorter drawing time at a higher temperature resulted in unsatisfactory toughness. This is one instance wherein drawing temperature - time gave some very interesting information.

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[1] Research Engineer, Scovill Manufacturing Co., Waterbury, Conn.

[2] Consulting Engineer, American Chain Co., Bridgeport, Conn.

[3] Chief Metallurgist, Henry Vogt Machine Co., Louisville, Ky.

This may be of interest in connection with the drawing of properly quenched high-speed steel. Several years ago our tool manufacturers learned that if the drawing time be considerably prolonged, a very beneficial effect was observed in the way of increased tool life with high-speed steel.

Grinding, being a "destructive" operation unless sufficient care is exercised, may result in a practical destruction of the usefulness of the part, and even when conducted under exacting technique is almost impossible to completely prevent some deleterious effects. In the preparation of test specimens, unless these grinding effects are properly taken into consideration, the test results may be somewhat misleading as far as the characteristics of a certain analysis are concerned.

In view of this I should like to ask the authors the following questions:

1. If any test specimens were ground after quenching and drawing, were these specimens given a second draw prior to testing?

2. What was the temperature of the quenching brine; also the brine concentration?

3. In a general way, what considerations were observed in the selection of the quenching temperature, that is, how far above the upper critical temperature?

4. What was the temperature of the quenching oil used for the high-speed quenching, since this is very important from the standpoint of uniform quenching?

5. What observations have been made regarding the impact properties of 11 to 13 per cent chromium, 0.10 per cent carbon material which has been quenched from the drawing heat in comparison with material allowed to cool slowly from the same temperature?

MESSRS. G. V. LUERSSEN[1] AND O. V. GREENE[1] (*authors' closure, by letter*).—Mr. Rawdon's question was at least partially answered by Mr. de Forest. We feel that the peak of maximum toughness found in drawing carbon tool steel is due to a fairly complete removal of residual stress and the slight agglomeration of the submicroscopic cementite and ferrite. The drop of impact value with hardness beyond the peaks as the drawing progresses is probably due to the mixed structure resulting from the transformation of the retained austenite. As soon as all the austenite is transformed, the curves again begin to rise.

Mr. Styri suggests that the structures are not entirely martensitic as stated in the paper after drawing at 350 F. (175 C.). Austenite in small amounts is undoubtedly present. Its existence is probably one reason for the drop in impact as the drawing progresses, as mentioned above. Microscopically, of course, our structures look like pure martensite. After the structures become entirely troostitic, the curve again rises.

In answer to Mr. Hurtgen's inquiry in regard to drawing time, we hope to bring out a very complete answer in a future paper. To date we are not in exact agreement with the information he has in regard to the effect of drawing time. It may be that we are not talking about exactly the same thing, particularly, type of service. That is to say, it has been found that increasing the drawing time moves the peak laterally to the left at the same magnitude. In other words, a specimen drawn at 425 F. (220 C.) for 15 minutes will show about the same value as a specimen drawn at 325 F. (165 C.) for 8 hr., while the specimen drawn at 425 F. (220 C.) for 8 hr. has a much lower impact value. A practical application of this information has been found in heading dies where the faces of the dies have been drawn for only a short interval at 425 to 450 F. (220 to 230 C.). Dies treated in this manner gave fairly satisfactory results. However, dies drawn all over in this temperature range for an extended period of time showed much poorer results. On the other hand, dies drawn all over at 325 to 350 F. (165 to 175 C.) gave the best results. Apparently there is a correct drawing time and temperature for tools of every size and analysis.

Our specimens were machined and finished in the ¼-in. section and polished and hardened, and the scale polished off the surface by means of No. 00 French emery before testing. No attempt was made to grind, since we were familiar with the variables that might be introduced by this method of preparation. The brine as used for quenching the carbon tool steel was 15 per cent and the oil used for quenching the high-speed steel was at room temperature. The quenching temperatures used have long been established as correct for these particular grades of steel.

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[1] Metallurgical Dept., Carpenter Steel Co., Reading, Pa.

We have no information in regard to the application of the torsion impact test on stainless steels. The low-carbon stainless irons do not lend themselves to this test. Sufficient information can be obtained from them by means of the standard impact tests. There is no reason, however, why the torsion impact test cannot be applied to the higher-carbon stainless steels of the cutlery grade.

The authors are indebted to Mr. Scott for his usual scholarly remarks. The use of the age-hardening phenomenon to explain the characteristics of curves found with high-speed steel is particularly interesting.

# AN "OVERNIGHT" TEST FOR DETERMINING ENDURANCE LIMIT

By H. F. Moore[1] and H. B. Wishart[2]

## Synopsis

Five or six fatigue specimens of a metal are provided. The Rockwell hardness of each is determined. The specimens are then placed in rotating-beam machines, and are subjected to approximately 1,400,000 cycles of stress, which requires about 15½ hours for machines running at 1500 r.p.m. The fatigue machines may be conveniently started in the late afternoon, and then the specimen can be removed early the next morning; hence the term "overnight" test. The stresses for the different specimens cover a range of values on both sides of the estimated endurance limit. After the 1,400,000 cycles of reversed flexure the specimens are removed from the machines and pulled as tension specimens, noting the tensile strength. If any of the specimens breaks before the 1,400,000 cycles its tensile strength is placed at zero. Making corrections proportional to the Rockwell hardnesses, the test data are plotted with stress applied for the period of 1,400,000 cycles as ordinates and tensile strength, after this period, as abscissas. The endurance limit is taken as the ordinate corresponding to the maximum abscissa of this curve.

The theory of this test is that below the endurance limit cycles of repeated flexure *increase* the endurance limit and presumably the tensile strength, while above the endurance limit cracks begin to develop and, if allowed to spread, reduce the tensile strength. A period of 1,400,000 cycles seems to be sufficient to develop such cracks for the metals tested, with the possible exception of duralumin.

This method has been checked against long-time fatigue tests for structural steel, cold-rolled steel, brass, monel metal, nickel-steel, chromium-nickel steel, duralumin, and specimens of cold-rolled steel with sharp notches. The maximum variation observed in endurance limit as determined by this "overnight" test and as determined by the long-time test was 11.8 per cent, for duralumin.

A chief obstacle to the development of "fatigue" testing of metals on a commercial basis is the long time required to make an accurate determination of the endurance limit, or the "fatigue" limit, as it is sometimes called. Numerous accelerated tests for endurance limit have been proposed, among them tests depending on inelastic action or on mechanical hysteresis, tests depending on rise of temperature of specimen, tests depending on the deflection

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[2] Graduate Student in Engineering Materials, University of Illinois, Urbana, Ill.

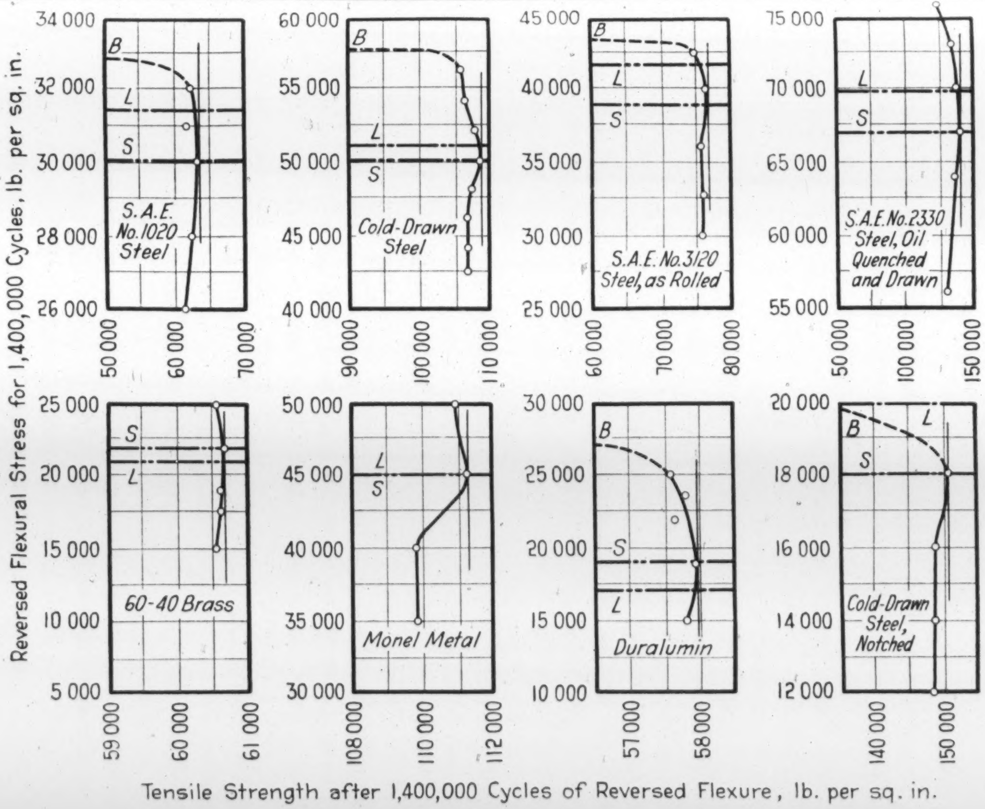

FIG. 1.—Data for "Overnight" Tests.

*L* denotes endurance limit by long-time test.
*S* denotes endurance limit by "overnight" test.
*B* gives stress at which specimen broke before 1,400,000 cycles of reversed flexure had been applied.

or other deformation of specimen as cycles of stress are applied, tests depending on the energy absorbed in applying cycles of stress, tests depending on changes of magnetic properties, and tests depending on change of electrical resistance as fatigue cracks developed in a specimen. None of these have proved reliable as a general fatigue test for metals.

In connection with the study of the spread of cracks in rail steel another somewhat accelerated test for endurance has been tried out by the authors, and, while such a test must be tried out by a number of laboratories before its usefulness can be regarded as generally established, this paper presents it to this Society in the hope that some laboratories may try it out further.

The theory on which this test is based is that under cycles of repeated stress metals are subjected to two opposing tendencies: (1) a tendency to increase of strength due, presumably, to cold work, and (2) a tendency to loss of strength due to the spread of fatigue damage by cracks or otherwise. Below the endurance limit the beneficial influence predominates, above the endurance limit the beneficial influence of cold work predominates for a time, but the fatigue cracks formed grow, at first slowly and then more and more rapidly,[1] and the destructive influences finally cause fracture, sometimes only after hundreds of millions of cycles of stress.

This general idea was embodied in the following test. Using five or six fatigue specimens, subject each of them to a number of cycles of a definite stress, but let the magnitude of the cyclic stress for different specimens vary from a value well below the estimated endurance limit for the least stressed specimen to a value well above the estimated endurance limit for the most heavily stressed. After the given number of cycles of stress have been applied remove the specimens from the fatigue testing machines and determine the tensile strength of each. For the specimens which have been subjected to cyclic stress below the endurance limit the tensile strength will either be increased or practically unaffected; for the specimens in which fatigue damage has made appreciable progress the tensile strength will be reduced. Plot a curve with magnitude of cyclic stress applied as ordinates and tensile strength after application of a given number of cycles of repeated stress as abscissas (see curves in Fig. 1). If the period of cyclic stressing has been sufficiently long to develop appreciable fatigue damage in those specimens which have been subjected to cyclic stress above the endurance limit, then the endurance limit is represented by the ordinate corresponding to the maximum abscissa.

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[1] For a study of increase of strength with cycles of stress below endurance limit, see *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 295 (1930); and for an illustration of the rate of spread of a fatigue crack see *Bulletin No. 165* of the University of Illinois Engineering Experiment Station, p. 20.

In trying out this test, rotating-beam fatigue machines of the "Farmer" or "Sondericker" type were used with specimens 13 in. long; however, short specimens could be used, such as are used in the R. R. Moore machine, or in the short specimen attachment of the Illinois-Hayes machine. The first series of tests was made using a period of cyclic stressing of 60,000 cycles, but this, while giving promising results, seemed too short a period, and a period of 1,400,000

TABLE I.—TYPICAL TEST DATA FOR "OVERNIGHT" TEST FOR ENDURANCE LIMIT.

Material: S.A.E. No. 1020 Steel, tested as rolled.

| SPECIMEN NUMBER | MAGNITUDE OF REVERSED FLEXURAL STRESS APPLIED FOR 1,400,000 CYCLES, LB. PER SQ. IN. | ROCKWELL HARDNESS NUMBER OF SPECIMEN, "B" SCALE | TENSILE STRENGTH OF SPECIMEN AFTER 1,400,000 CYCLES OF REVERSED FLEXURE, LB. PER SQ. IN. | |
|---|---|---|---|---|
| | | | AS TESTED | REDUCED TO ROCKWELL HARDNESS, "B" SCALE OF 60 |
| 3 | 26 000 | 60.0 | 61 500 | 61 500 |
| 6 | 28 000 | 61.2 | 62 400 | 61 200 |
| 13 | 30 000 | 60.0 | 63 200 | 63 200 |
| 16 | 31 000 | 60.0 | 62 000 | 62 000 |
| 19 | 32 000 | 60.2 | 62 600 | 62 300 |
| 14 | 33 000 | 61.2 | 0[a] | ..... |

[a] Specimen broke before 1,400,000 cycles of reversed flexure at 33,000 lb. per sq. in. had been completed.

Endurance limit by "overnight" test, 30,000 lb. per sq. in. (see also Fig. 1).

Endurance limit by "long-time" test, 31,500 lb. per sq. in. (see Table III).

TABLE II.—COMPARISON OF ENDURANCE LIMITS BY "OVERNIGHT" AND "LONG-TIME" FATIGUE TESTS.

Rotating-beam testing machine used at 1500 r.p.m.

| METAL | ENDURANCE LIMIT, LB. PER SQ. IN. | | DIFFERENCE IN PERCENTAGE OF "LONG-TIME" VALUE |
|---|---|---|---|
| | "LONG-TIME" TEST | "OVERNIGHT" TEST | |
| S.A.E. No. 1020 steel | 31 500 | 30 000 | 4.8 |
| Cold-drawn steel | 51 000 | 50 000 | 2.0 |
| S.A.E. No. 3120 steel, as rolled | 42 000 | 38 000 | 9.5 |
| S.A.E. No. 2330 steel, heat-treated[a] | 70 000 | 67 000 | 4.3 |
| 60–40 brass, as rolled | 21 000 | 22 000 | 4.8 |
| Monel metal, as rolled | 45 000 | 45 000 | 0 |
| Duralumin | 17 000[b] | 19 000 | 11.8 |
| Cold-drawn steel, notched | 20 000 | 18 000 | 10.0 |

[a] Oil quenched from 1500 F. (815 C.), drawn at 900 F. (480 C.).

[b] For 77,000,000 cycles of stress.

cycles was next tried. This proved fairly satisfactory, except possibly for duralumin, and required a period of about 15½ hr. at a speed of machine of 1500 r.p.m. This meant putting in the specimens the last thing in the afternoon, and taking them out almost the first thing the next morning, hence the proposed name "overnight" test. Table I gives a typical summary for one "overnight" test, and Fig. 1 gives graphically the test results for the eight series of tests which, so far, have been made and checked against long-time determinations of the endurance limits.

Table III.—Data of "Long-Time" Fatigue Tests.

| Specimen | Extreme Fiber Stress, lb. per sq. in. | Number of Cycles for Fracture | Endurance Limit, lb. per sq. in. | Specimen | Extreme Fiber Stress, lb. per sq. in. | Number of Cycles for Fracture | Endurance Limit, lb. per sq. in. |
|---|---|---|---|---|---|---|---|
| **S.A.E. No. 1020 Steel** | | | | **60–40 Brass** | | | |
| No. 5 | 35 000 | 388 000 | ...... | No. 9 | 25 000 | 6 019 600 | ...... |
| No. 8 | 33 000 | 675 600 | ...... | No. 11 | 23 000 | 11 341 800 | ...... |
| No. 14 | 33 000 | 1 392 100 | ...... | No. 8 | 22 000 | 18 401 700 | ...... |
| No. 18 | 31 500 | 20 000 000[a] | ...... | No. 10 | 21 000 | 130 354 000[a] | 21 000 |
| No. 11 | 30 000 | 19 300 000[a] | ...... | | | | |
| No. 2 | 28 600 | 27 870 000[a] | 31 500 | | | | |
| **Cold-Drawn Steel** | | | | **Monel Metal** | | | |
| No. 7 | 58 000 | 282 000 | ...... | No. 9 | 55 000 | 2 069 300 | ...... |
| No. 4 | 55 000 | 26 310 000 | ...... | No. 10 | 50 000 | 16 367 000 | ...... |
| No. 12 | 53 000 | 16 064 400 | ...... | No. 11 | 48 000 | 39 085 000 | ...... |
| No. 14 | 50 000 | 20 122 000[a] | ...... | No. 12 | 46 000 | 67 073 000 | 45 000[b] |
| No. 10 | 47 100 | 21 198 200[a] | 51 000 | | | | |
| **S.A.E. No. 3120 Steel, as Rolled** | | | | **Duralumin** | | | |
| No. 8-c | 48 000 | 477 900 | ...... | No. 2-D | 28 000 | 965 700 | ...... |
| No. 13-c | 44 000 | 1 243 000 | ...... | No. 12-D | 22 000 | 2 076 700 | ...... |
| No. 6-c | 42 000 | 41 313 600[a] | ...... | No. 11-D | 18 000 | 77 945 300 | 17 000[b] |
| No. 8-c | 39 000 | 38 154 100[a] | 42 000 | | | | |
| **S.A.E. No. 2330 Steel, Oil Quenched and Drawn** | | | | **Cold-Drawn Steel, Notched** | | | |
| No. 74 | 79 000 | 781 100 | ...... | No. 45 | 33 000 | 136 000 | ...... |
| No. 77 | 76 000 | 1 778 700 | ...... | No. 47 | 30 000 | 222 900 | ...... |
| No. 72 | 73 000 | 656 200 | ...... | No. 51 | 24 000 | 386 000 | ...... |
| No. 82 | 70 000 | 73 000 000[a] | ...... | No. 53 | 21 000 | 693 800 | ...... |
| No. 80 | 67 000 | 56 300 000[a] | 70 000 | No. 48 | 20 000 | 45 200 000[a] | ...... |
| | | | | No. 44 | 18 000 | 120 526 700[a] | 20 000 |

[a] Specimen did not fracture.
[b] Estimated.

Before offering this method for study and experiment at other laboratories, it was obviously necessary to check up endurance limits determined by this method for a fairly wide variety of metals with endurance limits determined by long-time tests. As will be noted in Fig. 1 and Table II, four ferrous metals and three non-ferrous metals were thus studied, and one ferrous metal using a specimen with a sharp V-notch. All the specimens of any one metal were from the same bar, but even so it seemed questionable whether the variations in tensile strength due to the application of 1,400,000 cycles of reversed flexure would be any greater than the variations in virgin tensile strength of the material along the bar. As a means of minimizing this source of error the following procedure was followed. Before applying the 1,400,000 cycles of reversed flexure, the Rockwell hardness, "B" scale, of each specimen was determined at a number of points on each side of the reduced section of the specimen, and the average value taken as the Rockwell "B" scale hardness of the specimen. The tensile strength of the specimen after the application of the 1,400,000 cycles of stress was then multiplied by the ratio of an assumed standard hardness for the specimens of the metal to the hardness of the particular specimen. The assumed standard hardness for the group was taken at approximately the minimum hardness of the specimens. This procedure involves the assumption that tensile strength is proportional to Rockwell "B" scale hardness, which seems a fairly satisfactory assumption for the narrow range of values involved.

In each case the endurance limit was determined by the "overnight" test *before* a determination was made by a long-time test.

Figure 1 and Table II show the endurance limits for the two methods. Except for the cases of duralumin and of brass, the "overnight" test gives results "on the safe side" of those obtained by the long-time test, and for the brass tested the difference in endurance limits is slight.

The "overnight" test may be criticized as not giving a sharply defined location for endurance limit, and because one bad result might give a false maximum point for the diagram. However, in all the metals tested there was a fairly well-defined "hump" to the curve, and the location of the maximum tensile strength was determinable within a fairly narrow range, a range not greater than ± 2000 lb. per sq. in. in any case. In this connection it should be remembered that for long-time test results the endurance limit probably is uncertain over a range of ± 500 lb. per sq. in. The maximum difference observed between "overnight" endurance limit and "long-time" endurance limit was 11.8 per cent for duralumin. It seems probable that for duralumin the application of 1,400,000 cycles of stress is not sufficient to develop fatigue damage far enough to reduce the tensile strength of the metal appreciably, and that some larger number of cycles would be necessary.

Several possible modifications of this test at once suggest themselves, and it may be that some of them will yield criteria more sensitive than is the change of tensile stress after the application of many cycles of stress. Measurements of ductility after a period of cyclic stressing, measurements of a combination of tensile strength and ductility, or tensile impact values might prove indexes of endurance limit as good as, or perhaps better than, the tensile strength.

One or two tests have been made in which, after the application of 1,400,000 cycles of stress, each specimen was subjected to 5000 cycles of stress of a given minimum value, the stress was then "stepped up" from this minimum to a higher value and another 5000 cycles applied, and this process repeated until the specimen broke. The maximum strength developed in this way is, of course, no direct measure of the endurance limit of the metal. A graph was then plotted using as ordinates magnitudes of reversed flexural stress applied for 1,400,000 cycles and, as abscissas, the total number of cycles applied before the "step up" test caused fracture. The graphs obtained so far are similar in general character to those shown in Fig. 1, and the method of locating endurance limit is also similar. Although a few preliminary results seem favorable, not enough work has as yet been done with this "step up" modification of the "overnight" test to determine its value.

It is obvious that the "overnight" test may be materially shortened, or the number of testing machines required may be reduced by the use of higher-speed machines than those running at 1500 r.p.m.[1]

As noted previously, it is felt that when such a test as this "overnight" test has been given a preliminary try out by a laboratory, it is a good policy to describe the test before a technical society so that it may be criticized, and so that other laboratories may be encouraged to try it out, and if it be found at all reliable, to develop modifications and improvements.

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[1] In this connection it may be of interest to note that in the Fatigue of Metals Laboratory at the University of Illinois, G. N. Krouse has developed a rotating cantilever machine driven by an air turbine and provided with forced lubrication. This machine has been used successfully at speeds as high as 27,000 r.p.m., and has been used on several series of tests at 10,000 r.p.m.

# DISCUSSION

**Mr. J. B. Kommers**[1] (*presented in written form*).—The efforts of Messrs. Moore and Wishart to develop a technique for determining the endurance limit of a material in a fairly short time is to be commended. The fact that the method depends upon actual endurance tests of specimens is an important point in its favor.

A glance at Fig. 1 of the paper shows that none of the curves shows an increase of endurance limit when subjected to stresses less than the endurance limit for 1,400,000 cycles. This is undoubtedly due to the fact that the number of cycles is not large enough to cause much strengthening. In my paper presented before the Society in 1930[2] I showed that for a cast iron which was very susceptible to strengthening by under-stressing, the increase of strength for 1,400,000 cycles was perhaps 7 per cent. Many experiments on under-stressing which I have made since 1930 show that 10 per cent of strengthening seems to be the upper limit for many metals. This being the case it seems to me that the method suggested by Moore and Wishart must depend mainly upon the idea that stresses above the endurance limit will cause a decrease in the tensile strength. These remarks are, therefore, an argument in favor of the method, because if there were very much strengthening due to under-stressing, the method would tend to give results for the endurance limit which would be too low.

It will be noted that the paper does not discuss any tests on cast iron. I hope the method will be tried out with this material. I have had occasion at various times to determine the endurance limits of cast irons when only a few specimens were available. The endurance limit for gray cast iron and also for malleable cast iron is usually close to 50 per cent of the tensile strength. My method has been to determine the tensile strength before making any endurance tests, and then subject the first specimen to a unit stress slightly higher than 50 per cent of the tensile strength. In this way I have succeeded in determining the endurance limit with as few as five specimens.

For materials like brass and duralumin, which usually require large numbers of cycles to determine the unit stress at which the *S*-*N* diagram becomes horizontal, there may be some question as to whether 1,400,000 cycles is sufficient to produce reliable results. Only experience with the method can answer this question satisfactorily.

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[1] Professor of Mechanics, Engineering College, University of Wisconsin, Madison, Wis.

[2] J. B. Kommers, "The Effect of Under-Stressing of Cast Iron and Open-Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

MR. R. E. PETERSON[1] (*by letter*).—The proposed test seems as though it might be of value because it has some logic back of it—something entirely missing in some of the correlation attempts to develop "short-time" fatigue tests.

During the past few years, I have from time to time studied the appearance of fractured fatigue test pieces with the idea of increasing the value of *post-mortem* examinations of broken machine parts. It has been found that the appearance of the fracture is a function of stress, material, degree of stress concentration, etc.—information of prime importance in determining why any particular machine did not give satisfactory service. This brings me to one objection which might be mentioned; the overnight test is destructive but does not allow normal fractures to develop over the entire stress range tested.

It would seem that a better way of shortening the time of testing is to increase the speed. Some mention is made of this in a footnote. The problem of increasing operating speed calls for a careful application of dynamic principles and bearing design, but there is no doubt but what satisfactory operation can be obtained. Tests have shown that the endurance limit is not affected by speed appreciably until very high speeds are reached.

With regard to the method used in the overnight test there may be several ways of accentuating the hump for laboratory study purposes. Davidenkoff used liquid air to embrittle cracked fatigue specimens and in that way could find a decreased static breaking load even before visual cracking appeared.

MR. R. L. TEMPLIN.[2]—I do not know whether it is an advantage or a disadvantage that the metal, duralumin, always comes out as the odd one in these investigations. The discrepancy noted in the case of duralumin might be still further accentuated if the fatigue results obtained on the long-time test had been carried out to 500 million cycles—the number of cycles that we normally carry out such tests—rather than to 75 million as used in this particular instance. You will find in the paper I am presenting before the Society[3] a statement that there is as yet no short-time fatigue test considered suitable for the light metals and alloys, and while there is an element of hope for such alloys in the present research, I still stand on that statement. We find quite marked differences in the fatigue curves for the different aluminum alloys, for example; and whereas we might get a result as shown by these authors in the case of duralumin, we might get quite discordant results in certain other alloys of aluminum. We would not admit, in spite of the popular ideas that are still prevalent, that any of the aluminum alloys crystallize under fatigue action, but now and then there is evidence that certain metallographic changes take place under fatigue action; and it would be rather hazardous in my opinion to attempt to define endurance limits for such materials by a short-time test such as offered by the present authors. We will certainly be very much interested in trying this scheme in our fatigue work and certainly hope that it can be used with some degree of success.

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[1] Manager, Mechanics Division, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[3] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," see p. 364.

MR. T. McL. JASPER.[1]—I think that the fundamental redeeming feature of this test is the adjustment of tensile strength value by the use of the Rockwell hardness machine. It is a very ingenious method of making the adjustment. With reference to the question of the direct-stress machine being more valuable, I am wondering whether the Rockwell hardness would serve so adequately if the whole cross-section through the specimen was involved in the fatigue test. Probably the best fatigue machine has been used for the adjustment of the tensile strength when using a surface hardness method.

MR. A. V. DE FOREST.[2]—This test is valuable, as any test is valuable, if it will reduce the uncertainty of fatigue testing and enable us to get a more rapid method in order to cover a larger range of individual test specimens, so as to find the variations to be expected in the fatigue range of any given material under any given heat treatment. The principal trouble with the use of fatigue data is that the results depend on such a very small number of samples as to be hardly representative of a commercial run of material, particularly in steel. An even more important result from this investigation will be greater illumination on the old question as to what happens under repeated loading of metals. I should like to inquire what are the views of Mr. Moore on what actually changes during the fatigue life of a test specimen which is going to break at around 50 million reversals? Is there a general change in structure which persists throughout the life of the specimen, or is there a purely local effect in which the general properties of the material as a whole are totally unaffected?

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[1] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[2] Consulting Engineer, American Chain Co., Inc., Bridgeport, Conn.

MR. E. H. DIX, JR.[1]—From the viewpoint of one who is attempting to develop new alloys, an accelerated test has great advantages. I think we may draw an analogy between corrosion testing and endurance testing; both require a long time before we get the final answer. However, accelerated corrosion tests are used, and although they are open to many pitfalls, they are used in many cases with advantage. I think if you are judging this test from the viewpoint of final results, you may be much more skeptical than if you consider it as a pilot test and I feel that perhaps in development of new alloys some such test as this will be of very great usefulness.

THE CHAIRMAN (*Mr. H. J. Gough*).[2]—May I be permitted to make a few remarks on this paper which I find so very interesting? When Hanson and I, some ten or twelve years ago, looked into fatigue from a metallographic point of view, we came definitely to the conclusion that hardening under cyclic stressing goes on quite irrespective of the fatigue limit, that is to say, it occurs both below and above the fatigue limit; in our report we made the prediction that, if specimens were subjected to ranges of stress inferior or superior to the fatigue limit, an increase in hardness would result. Directly arising from that prediction, Atchison, of England, made some tests (others were carried out at the Royal Aircraft Establishment) and the predicted increase in hardness was observed. About the same time, at the University of Illinois, the same increase in strength was found by tension tests; if my recollection is correct, Kommers reported the results referred to.

With regard to the general subject of the paper, a satisfactory form of "short-time" fatigue test, I suppose I have spent as much time on that particular problem as anybody; I have arrived definitely at the conclusion that no reliable form of short-time test known has yet been devised. It is, of course, impossible to say if the present authors have been more successful with the method reported in the paper; I feel bound to remark, however, that, after a close study of fatigue phenomena I, at present, see no fundamental reason why any short-time test can be *expected* to prove reliable. I make this remark because it seemed to be inferred that the fatigue limit marks the dividing line between two essentially different behaviors of the material under test. This *may* prove to be so, but it is certainly not known at the present time. I cannot accept that such a dividing line exists. I think that the general effect on the material is essentially the same at stress ranges inferior and superior to the endurance limit. In certain very local regions there is a limiting critical condition set up under an unsafe range of stress which ultimately results in the formation of a crack, and at present, I see no real reason why any short-time method should disclose the limiting range of stress.

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[1] Chief Metallurgist, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

I would also remind the authors that the effect of even visible cracks on such mechanical properties as the tensile strength is often so extremely small as to be difficult to reconcile with the mathematical theory of stress concentrations. At the same time I am fully aware of the fact that the authors merely bring forward their results and say: "Here is a method that has given us good results; try it on a large number of materials." The authors have obtained certain results which are in some accordance with the real endurance limit, and they invite everyone to look further into the efficacy of the method.

Another fact that leads one to suspect the reliability of any short-time method is this: In a research laboratory one often deals with materials of a high degree of uniformity; but commercial materials often show considerable variation, so that when a large amount of material has been carefully tested, the fatigue limit can only be recorded as lying between certain limiting values. If a short-time test is to be accepted it must be reliable, irrespective of the presence of local flaws and inhomogeneities which may not be present in all the specimens or be situated at the most highly stressed parts of those specimens. The experience I had personally with this practical aspect of fatigue testing was partly responsible for the somewhat critical and skeptical attitude I adopt towards "quick" methods of fatigue testing. The present method represents a compromise between a short-time test and an endurance test of the usual form. Its success depends upon the question whether 1,400,000 cycles is sufficient to develop a crack of such a size and shape as to overcome the hardening effect of the cyclic stresses. Many of us will watch this work with great interest in the hope that the authors will find time to look into it further using a much wider range of materials; I should like to suggest that they might pay some attention to "commercial" materials.

MR. E. E. THUM[1] (*by letter*).—Regardless of whether one agrees that Messrs. Moore and Wishart have a sound theoretical basis for their ingenious "overnight" test, their plan of "correcting" the tension tests according to hardness of the specimen is open to criticism, because the variation in hardness in a single bar of commercial metal is quite fortuitous. There is, therefore, no guarantee that the hardness

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[1] Editor, *Metal Progress*, American Society for Steel Treating, Cleveland, Ohio.

"determined at a number of points on each side of the reduced section of the specimen" represents the hardness of the metal at the ruptured zone should that specimen be pulled in tension, nor that it is a measure of the relative tensile strength of the reduced section. A better plan (still open to the same criticism, although to a lesser degree, despite the additional labor involved) would be to explore very carefully the surface hardness *at the reduced section* in slightly oversized samples which are then machined to exact dimension, thus removing the metal disturbed by the hardness tests.

All this, of course, dodges the issue that hardness, tensile strength, and the endurance properties themselves (as governed by the position of stress raisers and orientation of crystalline grains) may not be expressed numerically by a definite figure, but depend on the laws of probability and deviate about a mean value within a narrow or broad range depending upon the quality of the material and the testing technique.

MR. H. F. MOORE[1] (*author's closure, by letter*).—The various persons who have discussed this paper have nearly all emphasized the fact that before this "overnight" test can be regarded as reliable, it must be tried out and developed further. With this statement the authors of the paper are in hearty agreement. Mr. Peterson's suggestion of a tension test of a specimen cooled in liquid air is of interest, and I hope that his suggestion may be tried. The authors heartily agree with Mr. Templin's suggestion that aluminum alloys, and possibly some others, may require a "fatigue damage developing period" of more than 1,400,000 cycles.

Answering Mr. de Forest's question, we have found no evidence for any general structural change during a long-time fatigue test, but we do find the spread of a crack at an accelerated rate as the test proceeds. Sometimes more than one fatigue crack develops. It is the object of this "overnight" test to subject specimens to a sufficient number of cycles to develop fatigue damage (presumably in the form of a crack) sufficient to diminish the static tensile strength of the specimen.

Mr. Dix's suggestion that this test may prove a useful pilot test is gladly accepted by the authors. Mr. Gough points out that the hardening effect of repeated stress continues *above* the endurance limit as well as *below*. The authors certainly intended to recognize that fact. Apparently the *damaging* effect is either zero, or at most very slight, below the endurance limit, and hence the tensile strength of a specimen is increased. On the other hand, above the endurance limit the damaging effect tends to neutralize the strengthening effect, and, if the test be continued long enough, to reduce the actual strength of the specimen. The "overnight" test is an attempt to carry repeated stressing of a specimen to such a point that the tensile strength of the specimen will be materially reduced. The test is, of course, an arbitrary test, and can be justified only on the pragmatic conclusion that it *works* for a wide variety of materials. The tests quoted in this paper give a better agreement between long-time and accelerated endurance limit determinations for a wider variety of materials than has been shown by any other accelerated fatigue test with which the authors are familiar.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. Thum's criticism of the use of Rockwell hardness as an index of tensile strength is interesting, and the authors recognize that it has some weight. His suggested variation of the Rockwell test "correction" is interesting and worthy of trial. However, the authors point to the practical result that the use of the average Rockwell hardness near the reduced section did serve to give more definite curves of variation of tensile strength than were given by the use of uncorrected values. This again is a pragmatic justification. For each specimen ten determinations for Rockwell hardness were made.

# FATIGUE TESTS OF GALVANIZED WIRE UNDER PULSATING TENSILE STRESS[1]

By S. M. Shelton[2] and W. H. Swanger[3]

## Synopsis

The limiting range of pulsating tensile stress was determined at various mean tensile stresses on a heat-treated galvanized steel wire and on cold-drawn galvanized bridge wire of approximately the same carbon content and tensile strength. The determinations were made on a Haigh alternating-stress testing machine set up to subject the specimens to tensile stresses only. The specimens were unaltered sections of commercially galvanized wire 0.192 in. in diameter. A device was developed for gripping the specimens which eliminated fractures of the specimens in the grips.

The limiting range of pulsating stress or "tensile fatigue limit" was determined at mean stresses increasing by increments of approximately 20,000 lb. per sq. in. between 50,000 and 150,000 lb. per sq. in. The results for each mean stress were plotted on *S-N* diagrams, and the limiting ranges for the various mean stresses were plotted on a single diagram for each type of wire. The results were also tabulated.

The results showed that the limiting range of pulsating tensile stress was practically independent of the mean stress within the range investigated.

## Introduction

For wrought metals, the tensile properties of test coupons are generally accepted as a criterion of the tensile properties of the structural member. In many cases the fatigue properties of a metal determined on machined and polished test specimens are not in accordance with the fatigue properties of a structural member of that metal. Surface characteristics such as decarburized layers, machine-tool marks, notches and certain types of protective metallic coatings, have been shown by many investigators to cause the fatigue limit to be much lower than the fatigue limit as determined on test coupons.

Limitations of testing equipment prevent the general use of actual structural members as specimens for fatigue tests. However, wire is a widely used structural material that does lend itself to fatigue testing without the necessity for machining or other preparation of

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[1] Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

[2] Assistant Metallurgist, U. S. Bureau of Standards, Washington, D. C.

[3] Metallurgist, U. S. Bureau of Standards, Washington, D. C.

test specimens. One of the authors of this paper devised a method for fatigue testing of unmachined specimens of wire by the rotating-beam method and showed that the fatigue limit of machined-and-polished specimens of a heat-treated wire was about twice that of the unmachined galvanized wire.[1]

In actual service, wire is rarely used to withstand stresses similar to those applied in a rotating-beam fatigue test. Except when used as a spring, wire is generally subjected to tensile stresses which may be purely static, but which probably are fluctuating more often than is generally believed. It therefore seemed desirable to determine the endurance limits of wire in tension, particularly the safe or limiting ranges of tensile stress for various mean tensile stresses.

In 1925, Moore and Jasper[2] stated that "A field of experimentation which would seem to offer possibility of study would be tests of long tension specimens under cycles of stress varying from a slight to a maximum tension." The results of an investigation along these lines applied to wire were given by Pomp and Duckwitz in 1931.[3] Their tests were made chiefly to show the effects of heat treatment prior to drawing and of various amounts of cold drawing on the fatigue properties of steel wire of 0.43, 0.62 and 0.83 per cent carbon contents.

F. C. Lea[4] devised a method for the torsional fatigue testing of unmachined specimens of wire. His results showed that the torsional-fatigue properties of unmachined wires were markedly lower than those of machined-and-polished specimens of the same wire.

The results reported in this paper were obtained by subjecting unmachined specimens of galvanized cold-drawn bridge wire and of a galvanized heat-treated wire of approximately the same carbon content and tensile strength to ranges of pulsating tensile stress at various mean stresses.

## MATERIALS

The heat-treated wire specimens were selected at random from a large quantity of wire, prepared under commercial conditions, which is on hand at the Bureau of Standards. The cold-drawn wire specimens also were cut at random from a lot which had been delivered for use in a suspension bridge cable. This method of selection was used so that the test results would be representative of a commercial product as delivered to the purchaser, rather than of a specially selected or prepared lot of material.

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[1] S. M. Shelton, "Fatigue Testing of Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 204 (1931).

[2] H. F. Moore and T. M. Jasper, "An Investigation of the Fatigue of Metals," *Bulletin No. 152*, Engineering Experiment Station, University of Illinois, Urbana, Ill. (1925).

[3] Anton Pomp and C. A. Duckwitz, "Endurance Tests of Steel Wires Under Fluctuating Tensile Stresses," *Mitteilungen*, Kaiser-Wilhelm Institut für Eisenforschung zu Düsseldorf, Vol. 13, No. 175, p. 79 (1931).

[4] F. C. Lea, "Torsional Fatigue Tests of Cold-Drawn Wire," *Proceedings*, Inst. Mechanical Engrs., Vol. 120, p. 661 (1931).

Both types of wire had a nominal carbon content of 0.75 per cent and a nominal manganese content of 0.50 per cent. Analyses of specimens from the same lots, but not necessarily from the same coils as the test specimens, indicated that neither the carbon content nor the manganese content varied more than 0.05 per cent from the nominal.

As the final step in its manufacture, the heat-treated wire was drawn through one hole from No. 4 (0.232 in.) hot-rolled rod and then subjected to a heat treatment which produced approximately the same tensile strength as the cold-drawn wire. It was galvanized by hot-dipping, the coating being approximately 0.001 in. thick.

Table I.—Physical Properties of the Materials.

| Galvanized Wire | Tensile Strength, lb. per sq. in. | Elongation in 5 in., per cent | Rockwell Hardness Number, "C" Scale | Torsions on Specimen 75 Diameters in Length, Turns |
|---|---|---|---|---|
| Heat treated.................... | 220 000 to<br>230 000 | 6.1 | 42 | 11¼ |
| Cold drawn...................... | 220 000 to<br>230 000 | 6.8 | 41 | 13 |

The cold-drawn wire was drawn through five holes from a ⅜-in. hot-rolled "patented" rod and then galvanized by the hot-dip process. The coating on the test specimens was approximately 0.002 in. thick. The nominal diameter of both types of wire was 0.192 in. (No. 6).

The ordinary physical properties of the wires are given in Table I.

## Test Method

The fatigue tests were made on a Haigh alternating-stress testing machine.[1] This machine permits a pulsating tensile stress to be superimposed on an initial tensile stress on the specimens. The initial tension was applied to the specimen through a spring, the tension of which was regulated by a graduated nut. The initial tension on the specimen in terms of graduations on the nut was determined by a calibration specimen of the same wire used for the fatigue tests. The load-extension relations of the calibration specimen were determined with a Ewing extensometer on a 2-in. gage length and a tension testing machine. The calibration specimen with the extensometer attached was then transferred to the Haigh machine and the nut was turned up to produce extension of the specimen and the stresses corresponding to the graduations on the nut determined from the load-extension relations of the specimen.

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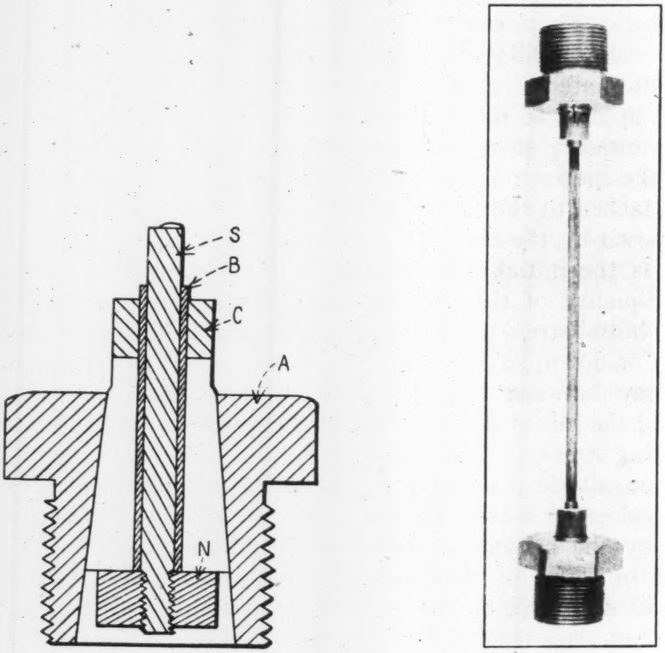
[1] A photograph of the Haigh machine used is shown, as set up for reversed stress tests on machined specimens, in a paper by R. D. France, "Endurance Testing of Steel: Comparison of Results Obtained with Rotating-Beam *Versus* Axially-Loaded Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 178 (1931).

The upper end of the specimen was attached to a fixed head on the machine. The lower end of the specimen was attached to an armature which oscillated between the pole pieces of two electromagnets alternately excited by a two-cycle generator. The amount of current applied to the magnets determined the magnitude of the range of pulsating stress on the specimen. The maximum tensile stress on the specimen is the sum of the initial stress imposed by the spring (attached to the armature) and half of the range of pulsating stress imposed by the magnets. The minimum tensile stress on the specimen is the initial stress minus half of the range of pulsating stress. Equality of the increments of pulsating stress above and below the initial stress was maintained by moving the specimen as a whole, up or down, so that the armature attached to the lower end was midway between the pole pieces. When this condition was maintained the initial stress set up on the specimen was the mean of the pulsating stress.

The magnitude of the ranges of the pulsating stresses used for the different values of mean stress was indicated on a "stress meter" actuated by the magnets. The readings of the stress meter were calibrated in terms of the load-extension relation of a calibration specimen of each type of wire as determined with a Ewing extensometer on a 2-in. gage length.

During the tests at the higher values of mean stress, the specimens took a permanent extension. This unbalanced the equality of oscillation of the armature on both sides of the midpoint between the pole pieces and lowered the initial stress imposed by the spring. By means of a hand-wheel provided on the machine the specimen was raised to bring the armature back to its original position, indicated by a zero reading of a differential ammeter when the current in both magnet circuits was equal. This also brought the initial stress imposed by the spring back to its original value.

It was necessary to readjust the load on each specimen at the beginning of a run because of the "give" in the grips and because of the extension of the specimen itself. This adjustment was generally completed during the first fifty thousand cycles of stress application, after which all of the specimens appeared to assume a cyclic state with no further permanent extension for the duration of the run. Naturally, when the specimen elongated, its diameter was reduced and the actual stresses, after the *loads* were brought back to their original values, were increased. It was found by actual measurement of the diameters of a number of permanently stretched specimens that the increase in stress due to reduced diameter was within the experimental error of the stress calculations, which is believed to be less than 5 per cent. Accordingly, the stresses recorded in the experiments reported below are those calculated on the original diameters of the specimens and the limiting ranges as given for the higher values of mean stress are on the "safe" side.



(*a*) Cross-sectional diagram of gripping device. Scale, actual size. *S*, specimen of wire; *B*, copper-lead alloy bushing: *C*, spring collet; *A*, threaded fitting attached to heads of machine; *N*, nut threaded on end of specimen.

(*b*) Specimen mounted in gripping devices. Reduction in diameter of specimen at polished ends is from 0.195 to 0.188 in. Over-all length, 10 in.

FIG. 1.—Gripping Device for Testing Specimen.

Since the thickness of the zinc coating of the cold-drawn wire was greater than the coating of the heat-treated wire, all stress calculations are based on the diameters of the steel wire specimens stripped of zinc in order to give a more nearly common basis for comparison.

The length of the specimens was 10 in. It was necessary to grip the ends in such a way that stress concentration due to pressure from the grip was at a minimum. A specimen mounted in the grips and a cross-sectional diagram of the gripping device are shown in Fig. 1.

A simple threaded fitting *A* was used to make the attachment to the dead and the live members of the machine. The tapered plug *C*, essentially a spring collet, was made by cutting four radial slots, 90 deg. apart, to within about ¼ in. from the smaller end. Parts *A* and *C* were made from machinery steel. The hole in the collet *C* through which the specimen extended, was drilled with a twist drill prior to cutting the slots. A nut *N* threaded on about ½ in. of the extreme end of the specimen was seated on the larger end of the collet. A pull exerted on the specimen *S* drew the collet through the tapered hole in *A* and caused it to grip the specimen. The specimen was thus held partly by the nut and partly by the friction between the specimen

TABLE II.—LIMITING RANGE OF PULSATING TENSILE STRESS AT VARIOUS MEAN STRESSES: HEAT-TREATED GALVANIZED WIRE.

| MEAN STRESS, LB. PER SQ. IN. | MAMIMUM STRESS, LB. PER SQ. IN. | MINIMUM STRESS, LB. PER SQ. IN. | LIMITING RANGE, LB. PER SQ. IN. | SEMI-RANGE, LB. PER SQ. IN. |
|---|---|---|---|---|
| 49 000 | 75 500 | 22 500 | 53 000 | ±26 500 |
| 68 000 | 92 500 | 43 500 | 49 000 | ±24 500 |
| 88 000 | 111 000 | 65 000 | 46 000 | ±23 000 |
| 107 000 | 130 500 | 83 500 | 47 000 | ±23 500 |
| 133 000 | 158 000 | 108 000 | 50 000 | ±25 000 |
| 153 000 | 177 000 | 129 000 | 48 000 | ±24 000 |

| ROTATING-BEAM TESTS | FATIGUE LIMIT, LB. PER SQ. IN. |
|---|---|
| Galvanized wire | 52 000 |
| Specimens machined from wire | 110 000 |

TABLE III.—LIMITING RANGE OF PULSATING TENSILE STRESS AT VARIOUS MEAN STRESSES: COLD-DRAWN GALVANIZED WIRE.

| MEAN STRESS, LB. PER SQ. IN. | MAXIMUM STRESS, LB. PER SQ. IN. | MINIMUM STRESS, LB. PER SQ. IN. | LIMITING RANGE, LB. PER SQ. IN. | SEMI-RANGE, LB. PER SQ. IN. |
|---|---|---|---|---|
| 50 000 | 80 000 | 20 000 | 60 000 | ±30 000 |
| 73 000 | 97 000 | 49 000 | 48 000 | ±24 000 |
| 90 000 | 114 000 | 66 000 | 48 000 | ±24 000 |
| 110 000 | 135 000 | 85 000 | 50 000 | ±25 000 |
| 135 000 | 156 500 | 113 500 | 43 000 | ±21 500 |
| 156 000 | 178 000 | 134 000 | 44 000 | ±22 000 |

| ROTATING-BEAM TESTS | FATIGUE LIMIT, LB. PER SQ. IN. |
|---|---|
| Specimens machined from wire | 118 000 |

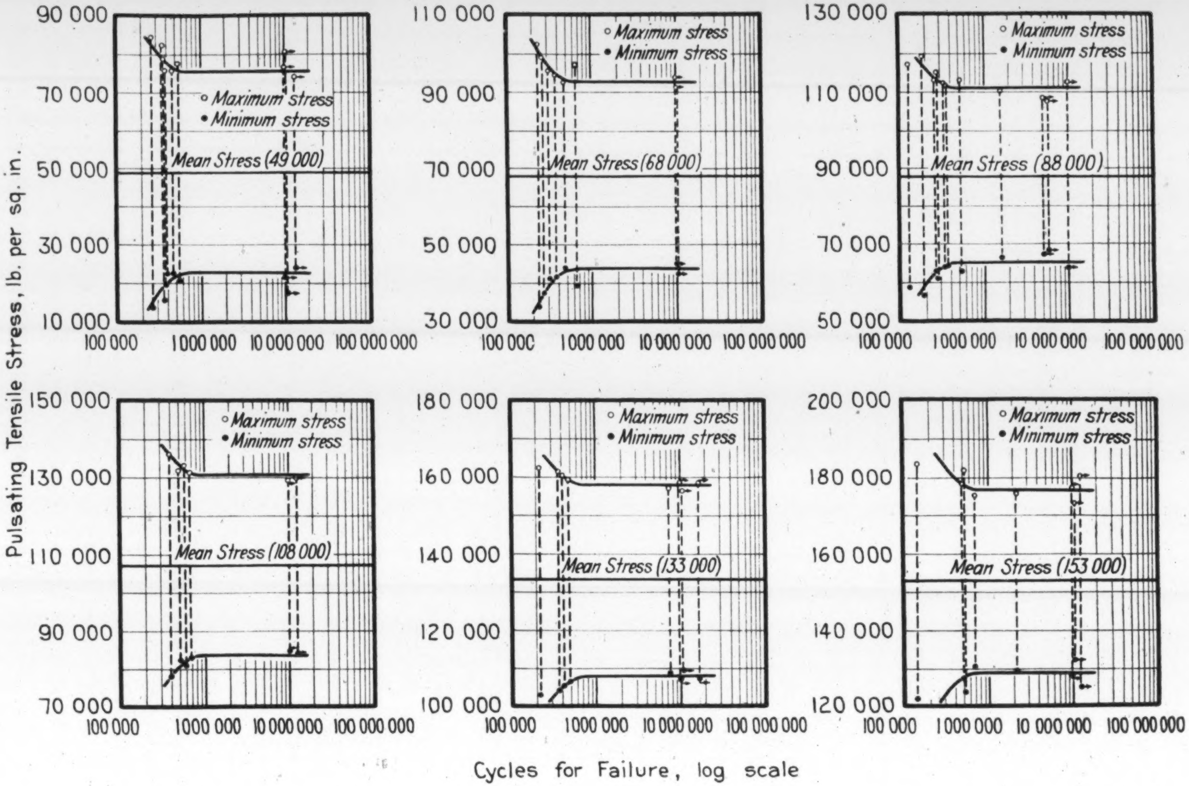


FIG. 2.—Relation Between Range of Pulsating Tensile Stress and Number of Cycles for Failure. Heat-treated galvanized bridge wire.

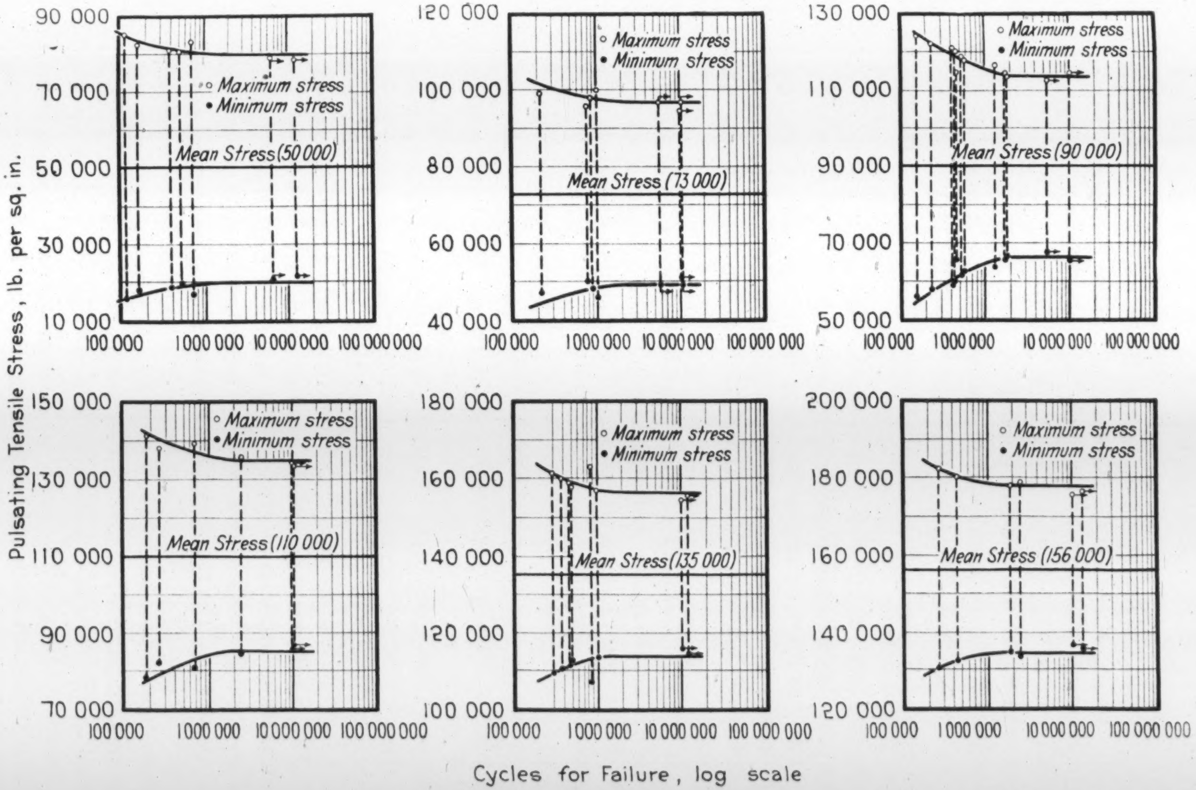


FIG. 3.—Relation Between Range of Pulsating Tensile Stress and Number of Cycles for Failure. Cold-drawn galvanized bridge wire.

and collet. The zinc coating on the ends of the specimens in the gripping device was ground off and the surface polished with 0000 emery paper. The diameter was reduced approximately 0.008 in. By this means stress concentration due to the presence of the galvanized coatings and pits in the wire was eliminated. To make the distribution of the stresses caused by the pressure of the collet on the specimen as uniform as possible, a soft metal bushing *B* was slipped over the polished end of the specimen. Copper and brass tubing were tried but proved to be unsatisfactory because both galled the steel surface of the specimen and frequently fatigue fractures radiated from the galled areas. This difficulty was practically eliminated with a bushing drilled from a rod of copper-lead bearing alloy containing approximately 25 per cent lead.

Fatigue limit determinations for both types of galvanized wire were made at increments of approximately 20,000 lb. per sq. in. mean stress, between mean stresses of 50,000 and 150,000 lb. per sq. in.

A sufficient number of specimens were run at each mean stress to insure a reasonable determination of the characteristics of the *S-N* diagram at that mean stress. The machine operated at a rate of 2400 cycles per minute.

## TEST RESULTS

Figure 2 shows the results obtained on the heat-treated wire and Figure 3 those for the cold-drawn wire. The results are also given in Tables II and III. Both the maximum and minimum stresses are represented on the diagrams as it is thought a clearer picture of the stress conditions will be obtained than if only the semi-range of stress were indicated as is usual for completely reversed stresses.

The limiting ranges of pulsating tensile stress for the various mean stresses are also plotted in Fig. 4 for the heat-treated wire and in Fig. 5 for the cold-drawn wire. The pulsating stresses are plotted as ordinates and the mean stresses as abscissas. The tensile strengths are also indicated on the diagrams. The fatigue limits for complete reversal as determined previously by rotating-beam tests on the heat-treated galvanized wire and on polished steel specimens machined from the wire are also shown in Fig. 4. It has not been possible to make rotating-beam tests on the galvanized cold-drawn wire because of the curvature of the wire imparted in drawing as it is coiled on the take-up blocks from the dies. The fatigue limit obtained by rotating-beam tests on short polished specimens machined from the cold-drawn wire is shown on the diagram, Fig. 5.

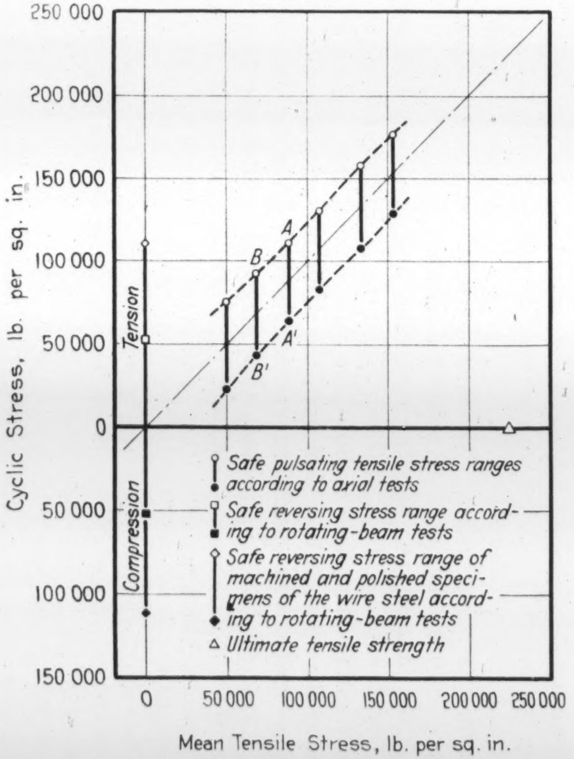


FIG. 4.—Limiting Ranges of Pulsating Tensile Stress at Various Mean Stresses. Heat-treated wire.

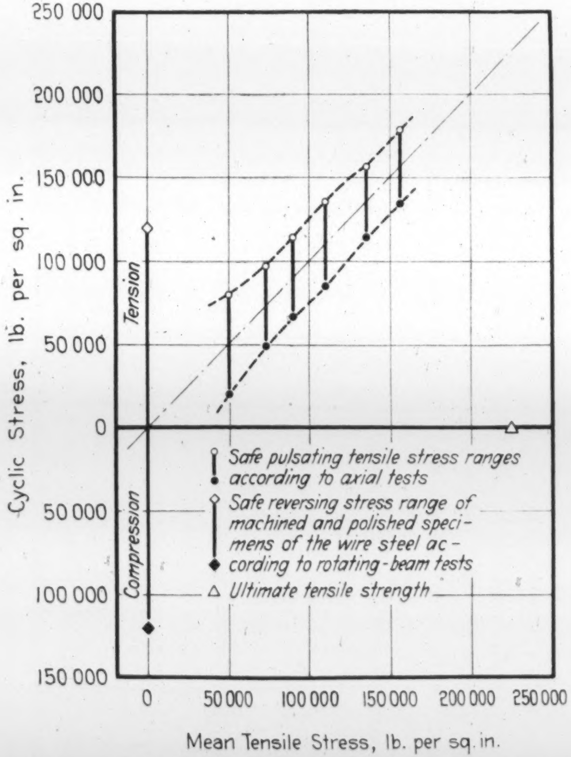


FIG. 5.—Limiting Ranges of Pulsating Tensile Stress at Various Mean Stresses. Cold-drawn wire.

## DISCUSSION

The results indicate, as is shown in Figs. 4 and 5, that the limiting range of tensile stress was not greatly different at the various mean stresses within the test range 50,000 to 150,000 lb. per sq. in. For the heat-treated wire the limiting range of pulsating tensile stress was 53,000 lb. per sq. in. at the lowest mean stress employed, whereas the average value of the limiting ranges, throughout the range of mean stresses, was 49,000 lb. per sq. in. The deviation from the average at the mean stresses from 68,000 to 153,000 lb. per sq. in. was probably within the experimental error. For the cold-drawn wire the limiting range of pulsating tensile stress was 60,000 lb. per sq. in. at the lowest mean stress but the average of the limiting ranges, was also 49,000 lb. per sq. in. However, the tendency for the limiting range to be less as the mean stress was increased is more pronounced in the cold-drawn than in the heat-treated wire. It was not practicable to make determinations of the limiting ranges of tensile stress on these wires at mean stresses much below the lowest value used, because it was necessary to maintain the minimum stress of the cycle at a value not much below the minimum used (10,000 lb. per sq. in.) to avoid introducing any stresses other than pure tension on the specimens. To extend the maximum and minimum stress curves of Fig. 4 and 5 downward to meet the fatigue limits determined by the rotating-beam tests would require an assumption that the fatigue limit under reversed tensile and compressive stresses is the same as that under reversed flexural stresses. On this assumption it would follow that for lower values of mean stress than were used in these tests the limiting range of stress would be greater.

If the lines representing the maximum and minimum stresses (Fig. 4) were extended to meet the values of the fatigue limit obtained in the rotating-beam test of the unmachined wire, they would be curved in the opposite direction from the curvature generally shown for lines representing maximum and minimum stresses at various mean stresses plotted on Goodman type diagrams.[1] However, these results were obtained on unaltered commercial galvanized wire with surface characteristics that undoubtedly introduced stress concentration.

Somewhat similar results were shown by Schulz and Buchholtz[2] in a discussion of the effects of notches on fatigue properties of a number of steels.

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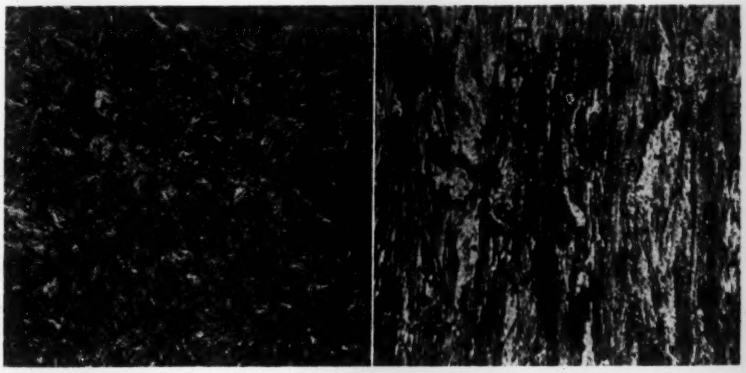
[1] H. F. Moore and J. B. Kommers, "The Fatigue of Metals," Chapter VII—The Effect of Range of Stress on Fatigue Strength, p. 173, McGraw-Hill Book Co., New York City (1927).

[2] E. H. Schulz and H. Buchholtz, "The Development of Fatigue Testing in Germany," *Proceedings*, Zurich Congress, Internat. Assn. Testing Mats., September 6 to 12, p. 293 (1931).

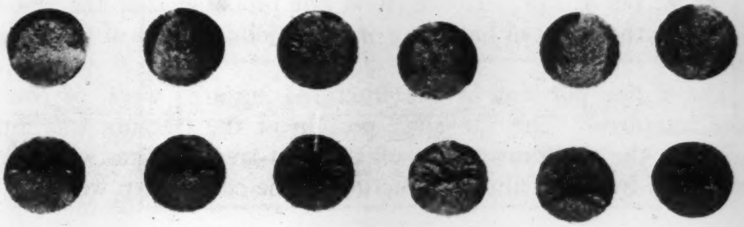
The capacity of the machine did not permit tests to be made on these wires at mean stresses higher than the maximum used (156,000 lb. per sq. in.). Hence, no indication was obtained of the course of the maximum and minimum stress curves between the highest value plotted and the point at which tensile fractures would be obtained.



(*a*) Structure of heat-treated wires, longitudinal section (× 500). Etched with 2 per cent nitric acid in alcohol.

(*b*) Structure of cold-drawn wire, longitudinal section (× 500). Etched with 2 per cent nitric acid in alcohol.

Heat-treated wire.



Cold-drawn wire.

(*c*) Fractured surfaces of specimens broken at each of the mean stresses, lowest mean stress at left (× 2).

FIG. 6.—Showing Structure and Fractured Surfaces of Heat-Treated and Cold-Drawn Wire.

The results obtained show definitely that the magnitude of the range of stress, rather than the value of the maximum stress, determined the fatigue limits of the materials. As an example of this fact, consider the limiting range *A*-*A*′, plotted on Fig. 4. The mean stress was 88,000 lb. per sq. in. The maximum stress was 111,000 lb. per sq. in. and the minimum stress was 65,000 lb. per sq. in. Suppose the minimum stress had been dropped to 25,000 lb. per sq. in., the maximum stress remaining unchanged. This would have brought the mean stress down to 68,000 lb. per sq. in., the mean stress at which the limiting range *B-B′* (Fig. 4) was determined. Figure 2 shows that at a mean stress of 68,000 lb. per sq. in. a specimen broke at approximately 230,000 cycles when the maximum stress was 102,000 lb. per sq. in. and the minimum stress was 34,000 lb. per sq. in. Consequently, a specimen stressed between 111,000 and 25,000 lb. per sq. in. would be expected to fail at a lower number of cycles. It is believed that this behavior of materials is frequently not considered in attempting to explain unexpected failures.

It is noteworthy that two materials with such a marked difference in metallographic structure should have such closely corresponding fatigue properties under the testing conditions used. The chemical composition and ultimate strength of both materials were nominally the same. Figure 6 (*a*) and (*b*) shows the structure of the two types of wire. Figure 6 (*c*) shows the appearance of the fracture of one specimen of each type of wire broken at each of the mean stresses used. The specimens are arranged in order of increasing mean stress from left to right.

The smooth areas nearest the circumference mark the extent of the "fatigue" part of the fractures. It is interesting to note that a smooth surface resulted even when the minimum stress was over 100,000 lb. per sq. in. Hence, it is not probable that the smooth surface was the result of battering of the adjoining faces of the fatigue crack.

The major portions of the fractured surfaces were, of course, tensile fractures. The "tensile" portion of the fracture was much rougher on the cold-drawn than on the heat-treated wires which is to be expected from the fibrous structure of the cold-drawn wire.

# DISCUSSION

**Mr. R. L. Templin.**[1]—We have had occasion to make fatigue tests to define the Goodman diagram of high-strength cold-drawn galvanized steel wire, with results shown in the accompanying Fig. 1. This wire normally has a tensile strength of a little better than 200,000 lb. per sq. in, a yield strength of about 170,000 lb. per sq. in., and 6 per cent elongation in 10 in. In Fig. 7 of the paper being presented by the writer[2] is shown a picture of the machine used in making such tests. The specimens are 50 ft. long; they are subjected to an initial tension and are then caused to vibrate so as to produce tensile stresses and compressive stresses, or different values for tensile stresses, in the outer fibers of the wire. I think the safe range of stress is somewhat higher than that indicated by the authors of this paper. It is, of course, perhaps a different type of steel wire than used in their tests.

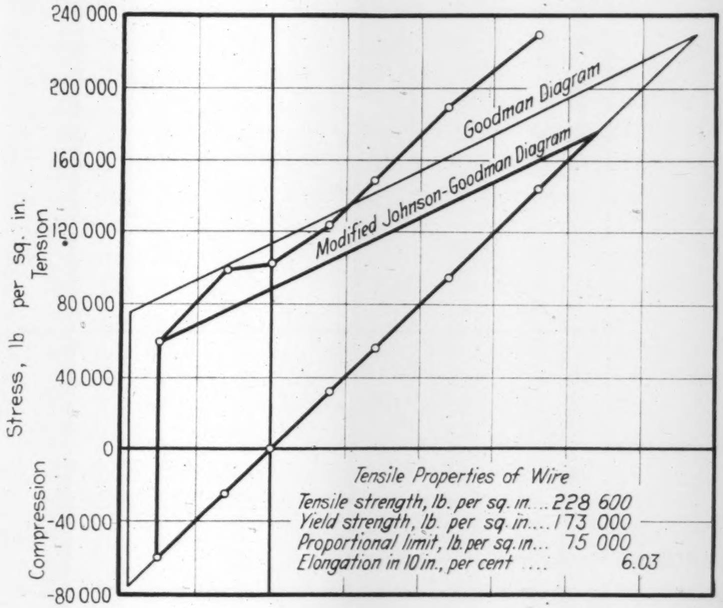


Fig. 1.—Range of Stress Diagram for 0.167-in. Galvanized Steel Wire.

**Mr. H. J. Gough.**[3]—I am very much interested in the very ingenious method of gripping the specimen described in this paper. An interesting and somewhat surprising result reported is the small effect on the safe range of stress of variations in the value of the mean stress. I do not agree that it is legitimate to take the fatigue values obtained from an axial loading machine and then include in the same diagram as directly comparable the data obtained from a flexural machine. I think this is not justifiable either theoretically or from the results of practical experience because the two types of results often do not check.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," see p. 364.

[3] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

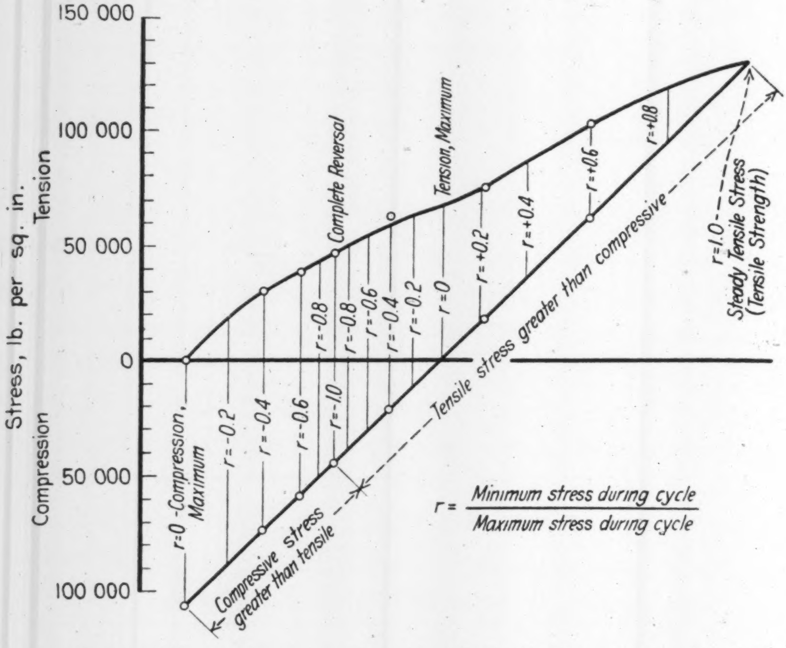

FIG. 2.—Goodman-Type Diagram—Showing effect of range of stress on fatigue strength of rail steel.

I should like to ask the authors what is the effect of galvanizing as such. Previous papers on the subject show that, in some cases, unless very special attention is paid to the method of galvanizing, severe internal stresses can be induced which reduce the safe range; by paying due attention to the method employed, these stresses can be reduced.

Discussing a point Mr. Templin raised, there is no reason why any experimental fatigue results should agree or disagree with the Goodman or Gerber relation; the Gerber relation was purely an experimental curve obtained under certain test conditions while the Goodman relation was based on a misunderstanding as to what fatigue action really is. If one were foolish enough, for a material in general, to design for a high range of stress, based on either of those formulas, one would be inviting trouble, as it is now clear that no *general* relation exists between mean stress and safe range of stress.

MR. H. F. MOORE.[1]—I should like to discuss this matter of range of stress, merely to add a little data on the subject. In connection with the investigation of fissures in rails, now in progress at Illinois, tests have been made to determine the range diagram for rail steel. An axial strength machine of the elastic-ring type was used. This machine was designed in the Fatigue of Metals Laboratory by Mr. Krouse and the writer. The diagram obtained at Illinois is shown in the accompanying Fig. 2. These data from our laboratory, like the data presented by Shelton and Swanger, show that not enough attention has been paid to the range of stress near the tensile ultimate, and also that neither the Gerber relation, nor the Goodman diagram, nor the modified Goodman diagram is to be regarded as giving any exact general relation between range of stress and endurance limit.

MR. W. H. SWANGER[2] *(author's closure)*.—In reply to Mr. Gough's remark concerning the results obtained with rotating-beam tests on the heat-treated steel wire, I wish to emphasize that they were placed on the diagram showing the results of the axial loading machine tests simply as information. The diagram was purposely not extended to meet the rotating-beam test values because we felt, in agreement with Mr. Gough, that it would not be safe to do so.

The effect of galvanizing, as such, on the endurance properties of this heat-treated wire under pulsating tensile stresses has not yet been determined. In a paper presented before the Society last year,[3] it was shown that in rotating-beam fatigue tests of machined specimens of 0.72 per cent carbon steel, quenched and tempered to produce a structure similar to that of the heat-treated wire, the fatigue limit of hot-dip galvanized specimens was 42 per cent lower than the fatigue limit of the polished ungalvanized specimens. The decrease was considered to be a combined effect of pickling and galvanizing. For specimens of this steel that were pickled but not galvanized the decrease was only 7.5 per cent. Although the results reported in the paper for other materials were not in exact agreement with these, it was found that specimens that had been galvanized and then stripped of their zinc coatings would "run" at stresses just under the endurance limits of the pickled specimens.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Metallurgist, U. S. Bureau of Standards, Washington, D. C.

[3] W. H. Swanger and R. D. France, "Effect of Zinc Coatings on the Endurance Properties of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 430 (1932)

# THE FATIGUE PROPERTIES OF LIGHT METALS AND ALLOYS

By R. L. Templin[1]

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## Synopsis

The paper includes a discussion of the various types of fatigue tests made on light metals and alloys together with a description of types of machines and apparatus used in tests made by the Aluminum Company of America. A new design of direct tension-compression fatigue machine is described and a brief description of the high-speed vibrating-wire type is also given.

A considerable amount of fatigue data is given for nearly all the commercial light alloys of aluminum, both wrought and cast, and for several magnesium-base alloys, as well as a discussion of the relation between endurance limit and tensile strength. The effects of cold working and heat treatment on the endurance limit are considered. Some data are given to show the effects of notches on the fatigue strength of certain alloys. Results obtained on different forms of product by means of different types of specimen and by various investigators are shown to give concordant results provided the tests are carried out to a sufficiently large number of cycles. That the number of cycles of stress required to define the endurance limits of most light alloys is of the order 500 million is shown by comparison with foreign laboratory test results, which generally show much higher endurance limits. Curves are given illustrating how misleading results may be obtained from tests not run a sufficiently large number of cycles.

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The extent to which government, university and commercial laboratories have gone in providing suitable equipment for making fatigue or endurance tests of metals is indeed material evidence of the need for more comprehensive knowledge of the strength of metals under various kinds and conditions of repeated stresses. From time to time there have appeared in the technical literature fatigue data pertinent to light metals and alloys but usually such data are obtained from tests of only one lot of material and one form of product. The reason for this meagerness of data is easily understood when consideration is given to the amount of time, equipment and expense necessary for carrying out fatigue tests. Unfortunately, as yet, no satisfactory "short-time" fatigue test has been found for the light metals and alloys. Furthermore, the testing machines, specimens, and methods used today in making fatigue tests have not been generally standardized with the result that certain variations are likely to occur in the data obtained, depending on these factors.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

From a consideration of these few facts, many questions arise concerning the accuracy of fatigue data; the effects of form and size of product; the influence of processes of fabrication; the relation of one type of fatigue test to another; the relation of fatigue strength to other mechanical properties; the comparison of the fatigue strengths of different metals; and many others of perhaps equal importance. While all of these cannot be answered fully at this time, yet results have been obtained from the tests carried out in the Aluminum Research Laboratories which, together with those from other sources, are at least indicative of the answers to some of the questions.

## TYPES OF FATIGUE TESTS

Various types of fatigue tests are carried out in the Aluminum Research Laboratories and they may be enumerated briefly as follows:

1. Rotating beam.
   (*a*) R. R. Moore type—various sizes of specimens.
   (*b*) Wire or small tubing of the Bureau of Standards type.
   (*c*) High-temperature short-specimen rotating beam.
2. Repeated flexure, for sheet, using constant deflection and triangular-shaped uniform-stress specimens.
3. Direct tension and compression.
4. Combined tension and bending type for wire and cable.
5. Corrosion-fatigue using repeated-flexure type machines.
6. Repeated impact, Stanton type.
7. Special repeated bending for small tubing and fittings (after H. F. Moore).

The R. R. Moore type of rotating-beam testing machine has been described elsewhere[1] so no further description of that machine will be given at this time. A battery of forty of these machines is used in the Aluminum Research Laboratories. In addition a much larger machine of the same general type has been provided which is arranged to take four different sizes of specimens, all geometrically similar to the usual R. R. Moore short type of specimen. These larger specimens have nominal diameters of $\frac{5}{8}$ in., 1 in., $1\frac{3}{8}$ in. and 2 in. In this machine the load is applied to the specimen by means of a calibrated coil spring and both spindles of the machine are driven from a jack shaft using V-type belts. The specimen is tested at a speed of 1400 r.p.m.

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[1] Report of Research Committee on Fatigue of Metals, *Proceedings*, Am. Soc. Testing Mats., **Vol. 30, Part I, p.** 259 (1930).

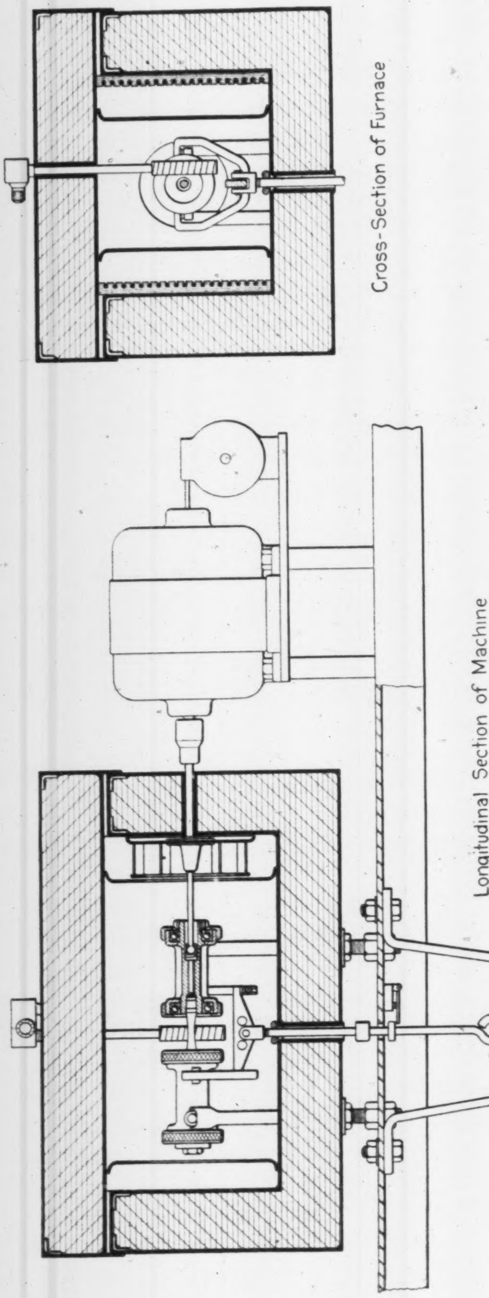
Cross-Section of Furnace
Longitudinal Section of Machine

FIG. 1.—High-Temperature Fatigue Testing Machine of a Short-Specimen Rotating-Beam Type.

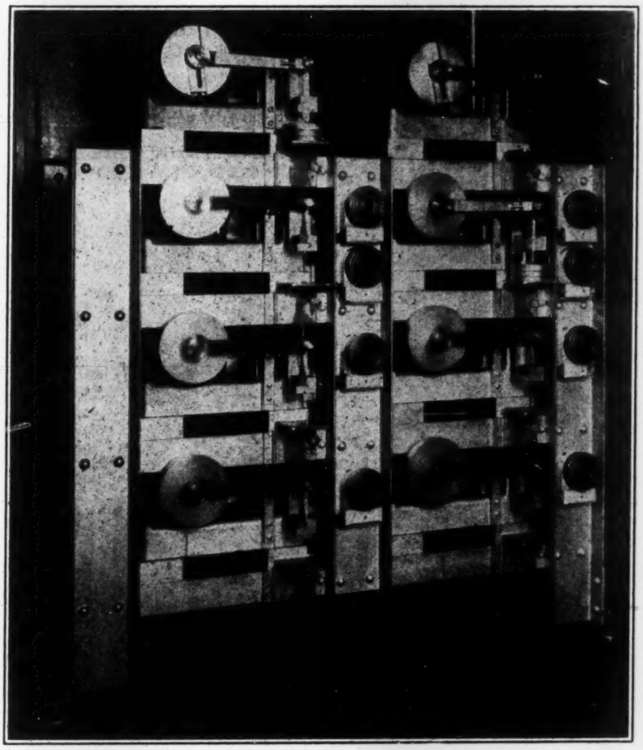

FIG. 2.—Repeated-Flexure Fatigue Testing Machine for Sheet Metal with Which Corrosion-Fatigue Tests May be Carried Out.

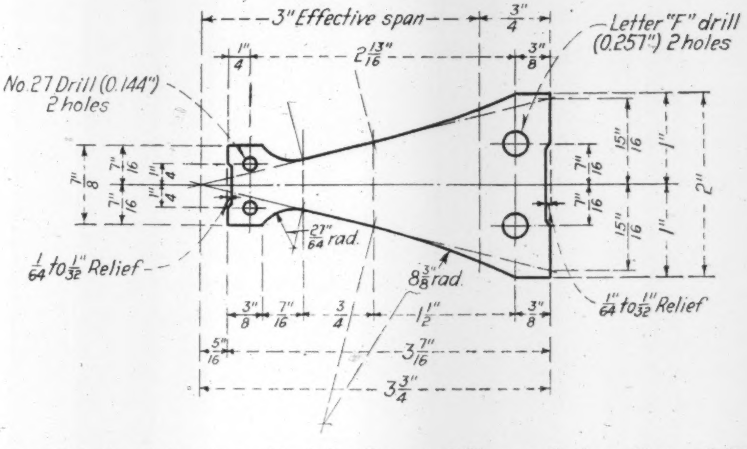
3" Effective span
3"/4
Letter "F" drill (0.257") 2 holes
1"/4
2 13"/16
3"/8
No. 27 Drill (0.144") 2 holes
7"/8
7"/16
7"/16
1"/4
1"/4
27"/64 rad.
1/64 to 1/32" Relief
8 3"/8 rad.
15"/16
15"/16
7"/16
7"/16
1"
1"
2"
1"/64 to 1"/32 Relief
3"/8
7"/16
3/4
1 1"/2
3"/8
5"/16
3 7"/16
3 3"/4

FIG. 3.—Details of Specimen Used for Repeated-Flexure Fatigue Tests of Sheet Metals.

Another type of rotating-beam machine used is a modified form of the type described by Shelton,[1] differing in many of its minor details, yet of the same general type. This machine is used for tests of wire and small tubing specimens having a maximum diameter of about $\frac{5}{16}$ in. and a length of 7 ft. The machine operates at various speeds up to about 3000 r.p.m.

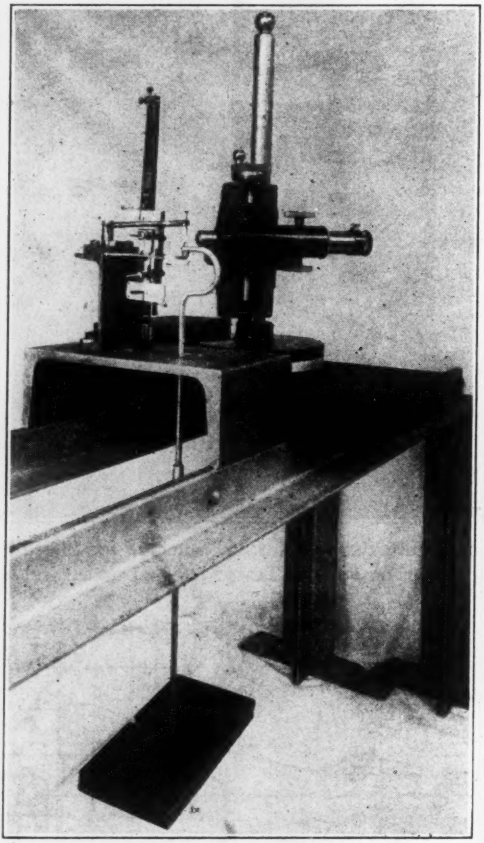

FIG. 4.—Calibration Set-up Used for Checking Stress and Deflection of Sheet Fatigue Specimens.

A high-temperature rotating-beam type machine has been designed and built but so far its use has not been of sufficient duration to warrant any conclusions being drawn from the results obtained. The machine is designed to operate at a maximum temperature of 600 F. (315 C.), using ball-bearing housings with the expectation of having to replace them frequently. The general drawing is shown in Fig. 1. It will be observed that the machine is of the short-specimen type with provision made to heat and circulate the air contained within the insulated box surrounding the machine. The temperature control is maintained by using a "Cenco" DeKhotinsky thermo-regulator. At the present time this machine is operating at a speed of 1800 r.p.m.

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[1] S. M. Shelton, "Fatigue Testing of Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 204 (1931).

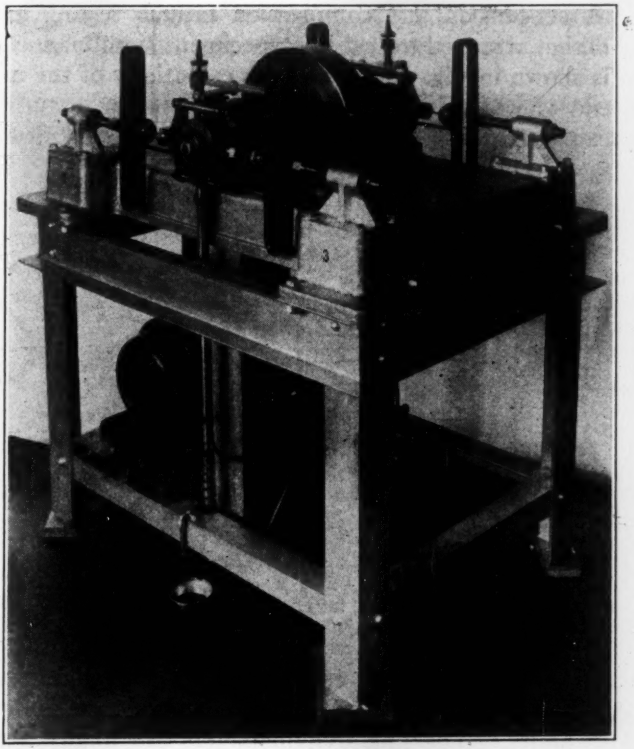

FIG. 5.—Direct-Tension and Compression Fatigue Testing Machine Accommodating Four Specimens Simultaneously.

Fatigue machines of the repeated-flexure type for testing sheet are shown in Fig. 2. These machines may be used simply as repeated bending fatigue testing machines or as corrosion-fatigue machines. The specimen used in these machines is shown in Fig. 3 and is quite similar to that previously used in the laboratories of the Goodyear-Zeppelin Corp. It is so proportioned that a considerable part of its length is subjected to uniform bending stress. The deflection of the specimen can be adjusted to within 0.001 in. by means of a graduated screw which is used to change the throw of the crank. These machines are of the constant deflection type and the stresses in the specimens have been checked not only by computations but also by actual calibration, using $\frac{1}{2}$-in. Huggenberger tensometers, supplemented with a compound filar microscope for measuring the deflection simultaneously with the stress determinations. The set-up used in this calibration work is shown in Fig. 4.

The direct-tension and compression fatigue testing machine is of a new design arranged to test four specimens simultaneously. This machine is shown in Fig. 5, and consists essentially of the main shaft on each end of which is a variable eccentric which in turn actuates a cross-head. To these cross-heads are attached one end of each of the specimens. The opposite ends of the specimens are attached to link dynamometers whose load-deflection curves are known within very close limits. The link dynamometers are finally attached to suitable brackets on the testing machine base. Adjustment of the graduated eccentrics determines the throw of the cross-heads, as well as the total range of stress for each pair of specimens. The throw of the eccentric may be varied from zero to $\frac{5}{8}$ in. Each of the links requires a load of 1000 lb. to cause a deflection of 0.129 in. The deflection of the links is measured on opposite sides at the center using a dial gage reading directly to the nearest 0.001 in. Adjustment of the nuts on the opposite sides of each bracket which supports a link affords a means for positioning the stress range of each specimen independently of the other specimens. That is, with a given stress range, by means of these adjusting nuts, all or any portion of the

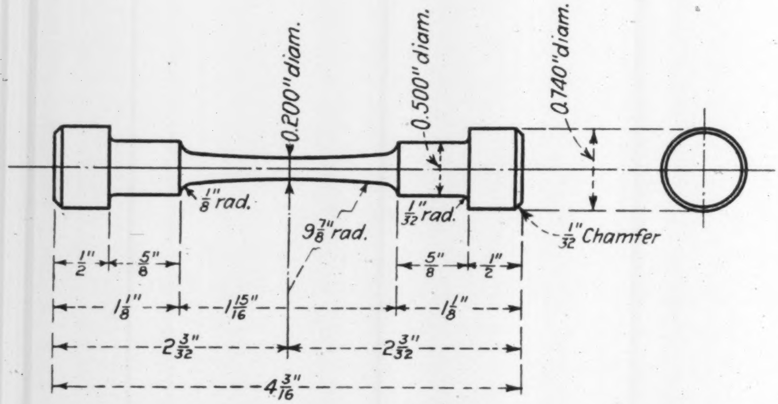

FIG. 6.—Details of Specimen Used in Direct Tension and Compression Fatigue Tests.

stress range may be made to cause either tensile or compressive stress in the specimen. Consideration of the machine just described will reveal that by adjustment of the throw of the eccentric and the diameter of the specimen, many different ranges of stress, as well as positions of ranges, can be obtained. An individual counter is provided for each specimen which automatically ceases to register upon fracture of the specimen. The machine is operated at a speed of 2000 r.p.m. The stresses in the individual specimens have been checked using $\frac{1}{2}$-in. Huggenberger tensometers on opposite sides of the specimens in a vertical plane and found to agree within less than 0.5 per cent. The details of the specimen generally used are shown in Fig. 6.

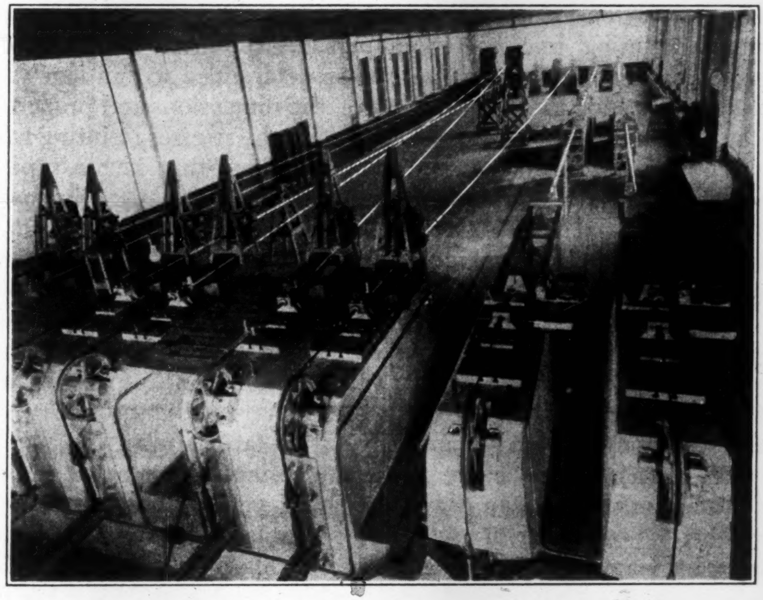

FIG. 7.—Fatigue Apparatus for Vibrating Tests of Small Wires and Cables.

The combined tension and bending type of fatigue machines used for wires and small cables are shown in Fig. 7 (the four spans on the right). These machines consist essentially of large concrete piers so arranged that a specimen 50 ft. long may be strung between them and maintained at a constant tension by means of suitable levers and dead weight. On the initial tensions thus put in the specimens, bending stresses are superimposed by means of an eccentric flywheel mounted directly on the specimen. This flywheel is rotated at speeds varying from a few hundred to 7200 r.p.m., depending upon the initial tension, the loop length used and the unit weight of the specimen. The unbalanced component of the flywheel is resisted in a horizontal plane by means of slender guide wires so that the specimens vibrate in a vertical plane only. During a test the specimen vibrates in a series of loops whose amplitudes are carefully controlled. The point of maximum stress, of course, occurs at the fixed end of the span where the specimen is rigidly clamped in a suitable fixture. This fixture however is free to move longitudinally on rollers. The deflection of the first loop of the specimen at this end of the span is determined by actual measurement to a suitable reference plane using micrometer calipers. Experience has definitely shown that this deflection curve can be determined within less than 0.002 in. Knowing the deflection curve, the dimensions and properties of the specimen, it is possible to determine the stresses resulting from the bending. In passing, it may be pointed out that such a type of machine and test is extremely sensitive to small variations in speed, power supply and temperature. On the other hand, it does afford a high-speed means of making combined tension and bending fatigue tests and with proper attention to details, proves quite satisfactory.

The corrosion-fatigue type of testing machine used has already been referred to, but it may be pointed out that so far, the actual tests made have been confined to studies wherein the specimen was constantly submerged in a $1N$ salt solution. In Fig. 2, only two of the machines are shown set up for corrosion-fatigue tests.

While many tests have been made using the repeated impact (Stanton) type of fatigue testing machine, yet because results from the other types appear to give more desirable information, there has been a tendency to discontinue the use of this type of fatigue testing machine for reasons pointed out by McAdam[1] and Moore.[2] The details of this machine are well known and therefore need no further comments at this time.

In addition to the types of fatigue machines just described, a special type, patterned after one of the designs developed by Moore, Kommers and Jasper,[3] is used for making fatigue tests of small-diameter tubing assembled with fittings of various types. In this machine arrangements are made so that the specimen is subjected internally to a low air pressure of about 20 lb. per sq. in. during test.

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[1] D. J. McAdam, Jr., "Endurance Properties of Steel: Their Relation to Other Physical Properties and to Chemical Composition," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 56 (1923).

[2] R. R. Moore, "Resistance of Metals to Repeated Static and Impact Stresses," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 547 (1924).

[3] H. F. Moore, J. B. Kommers, and T. M. Jasper, "Fatigue or Progressive Failure of Metals Under Repeated Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 266 (1922), Fig. 5.

TABLE I.—MECHANICAL PROPERTIES OF COMMERCIAL WROUGHT ALUMINUM ALLOYS.

| Alloy and Temper[a] | Size, in. | Form | Yield Strength (Set = 0.2 per cent), lb. per sq. in. | Tensile Strength, lb. per sq. in. | Elongation in Four Diameters, per cent | Reduct of Area, per cent | Brinell Hardness Number, 500-kg. load, 10-mm. ball | Shearing Strength, lb. per sq. in. | Endurance Limit, lb. per sq. in. (500,000,000 cycles) | Ratio, Endurance Limit to Tensile Strength |
|---|---|---|---|---|---|---|---|---|---|---|
| 2SO..... | 1⅛ | Rolled rod.................. | 3 150 | 12 600 | 41.5 | 81.0 | 21 | 9 600 | 5 000[d] | 0.40 |
| 2S½H .. | 1⅛ | Rolled rod.................. | 15 100 | 16 400 | 19.5 | 74.0 | 32 | 10 600 | 7 000 | 0.43 |
| 2S¾H .... | 1⅛ | Rolled rod.................. | 18 900 | 20 800 | 15.0 | 64.5 | 38 | 12 100 | 8 500 | 0.41 |
| | | | | | | | | | | |
| 3SO ...... | 1⅛ | Rolled rod.................. | 5 450 | 16 900 | 36.5 | 69.0 | 28 | 11 900 | 7 000[d] | 0.42 |
| 3S¾H .... | 1⅛ | Rolled rod.................. | 22 750 | 23 900 | 16.0 | 57.0 | 45 | 15 000 | 9 500 | 0.40 |
| 3SH...... | 1⅛ | Rolled rod.................. | 28 500 | 31 700 | 11.0 | 48.5 | 55 | 17 500 | 10 000 | 0.32 |
| | | | | | | | | | | |
| 4SO...... | ½ | Extruded and drawn rod.... | 10 000 | 25 350 | 23.0 | 58.7 | 46 | 16 500 | 14 000[d] | 0.55 |
| 4S¾H .... | ½ | Extruded and drawn rod.... | 36 500 | 39 300 | 6.5 | 41.4 | 72 | 21 000 | 15 000 | 0.38 |
| | | | | | | | | | | |
| 43SO..... | ¾ | Rolled and drawn rod ...... | 5 250 | 15 150 | 34.0 | 56.9 | 24 | 11 000 | 6 000 | 0.40 |
| 43SH..... | ¾ | Rolled and drawn rod ...... | 22 800 | 27 150 | 5.75 | 12.6 | 46 | 15 800 | 8 000[e] | 0.29 |
| | | | | | | | | | | |
| 14SW[b] ... | ¾ | Forged slab................ | 37 000 | 60 000 | 24.3 | 34.1 | 109 | 41 200 | 15 000[e] | 0.25 |
| 14SW[c] ... | ¾ | Forged slab................ | 38 000 | 62 000 | 21.3 | 28.1 | .... | 42 200 | 17 000[e] | 0.27 |
| 14ST[b].... | ¾ | Forged slab................ | 57 000 | 67 400 | 15.8 | 30.0 | 132 | 44 100 | 15 000 | 0.22 |
| 14ST[c]... | ¾ | Forged slab................ | 62 000 | 68 700 | 10.3 | 20.4 | .... | 43 200 | 17 000 | 0.25 |
| | | | | | | | | | | |
| 17SO..... | ¾ | Extruded and drawn rod.... | 9 800 | 25 250 | 23.0 | 48.5 | 44 | 18 100 | 11 000 | 0.44 |
| 17ST..... | ¾ | Extruded and drawn rod.... | 36 000 | 61 050 | 21.5 | 27.0 | 101 | 39 400 | 15 000 | 0.25 |
| 17ST..... | 1⅛ | Rolled rod.................. | 34 000 | 61 900 | 24.0 | 38.0 | 106 | 39 600 | 15 000 | 0.24 |
| | | | | | | | | | | |
| A17ST ... | ¾ | Extruded and drawn rod.... | 23 500 | 46 200 | 30.0 | 53.0 | 76 | 30 600 | 13 500 | 0.33 |
| B17ST ... | ¾ | Extruded and drawn rod.... | 29 000 | 52 900 | 28.0 | 50.0 | 86 | 33 900 | 15 000 | 0.29 |
| C17ST ... | ¾ | Extruded and drawn rod.... | 55 000 | 66 000 | 15.5 | 29.2 | 121 | 43 500 | 13 500 | 0.20 |
| | | | | | | | | | | |
| 24ST..... | ½ | Extruded and drawn rod.... | 49 000 | 70 000 | 18.3 | 33.2 | 108 | 41 000 | 14 000 | 0.20 |
| 24SRT ... | ½ | Extruded and drawn rod.... | 68 700 | 75 200 | 13.3 | 27.8 | 113 | 44 600 | 14 500 | 0.19 |
| | | | | | | | | | | |
| 25SO..... | ¾ | Extruded and drawn rod.... | 8 500 | 24 900 | 24.0 | 50.5 | 41 | 16 300 | 9 000 | 0.36 |
| 25SW .... | 1⅛ | Rolled rod.................. | 27 000 | 51 400 | 26.0 | 46.0 | 85 | 32 300 | 14 500 | 0.28 |
| 25ST..... | 1⅛ | Rolled rod.................. | 38 500 | 60 200 | 20.5 | 37.0 | 108 | 39 600 | 15 000 | 0.25 |
| 25ST..... | ¾ | Extruded and drawn rod.... | 31 500 | 58 600 | 27.0 | 46.0 | 100 | 37 350 | 15 000 | 0.26 |
| 25ST[b].... | ¾ | Forged slab............ | 34 200 | 54 540 | 22.0 | 37.8 | 100 | 38 000 | 14 000 | 0.26 |
| 25ST[c].... | ¾ | Forged slab................ | 38 100 | 58 850 | 12.0 | 12.3 | .... | 38 000 | 13 500 | 0.23 |
| | | | | | | | | | | |
| 32ST[b].... | ¾ | Forged slab................ | 47 000 | 54 500 | 7.70 | 10.0 | 126 | 37 800 | 14 000[e] | 0.26 |
| 32ST[c].... | ¾ | Forged slab................ | 46 000 | 51 600 | 3.85 | 6.1 | .... | 38 100 | 14 000[e] | 0.27 |
| | | | | | | | | | | |
| 51SO..... | 1⅛ | Rolled rod.................. | 5 500 | 15 800 | 33.0 | 68.5 | 27 | 11 000 | 6 500[d] | 0.41 |
| 51SW .... | 1⅛ | Rolled rod.................. | 18 500 | 36 600 | 29.5 | 50.5 | 62 | 23 500 | 10 500 | 0.29 |
| 51ST..... | 1⅛ | Rolled rod.................. | 35 000 | 45 500 | 13.5 | 19.0 | 91 | 29 600 | 10 500 | 0.23 |
| | | | | | | | | | | |
| A51ST[b] .. | ¾ | Forged slab................ | 38 100 | 46 700 | 22.0 | 37.5 | 99 | 33 600 | 11 000 | 0.24 |
| A51ST[c] .. | ¾ | Forged slab................ | 39 800 | 47 700 | 8.50 | 14.2 | .... | 31 900 | 10 000[e] | 0.21 |
| | | | | | | | | | | |
| 53SO..... | ¾ | Rolled and drawn rod....... | 5 650 | 14 900 | 39.0 | 67.7 | 24 | 10 450 | 7 000[e] | 0.47 |
| 53ST..... | ¾ | Rolled and drawn rod....... | 27 400 | 36 100 | 23.8 | 55.6 | 74 | 24 500 | 12 000[e] | 0.33 |

[a] O=annealed. H=hard. W=as quenched. T=heat treated. RT=cold worked after heat treatment.
[b] Longitudinal specimens.
[c] Transverse specimens.
[d] No fracture at any stress.
[e] Estimated from incomplete tests.

TABLE II.—MECHANICAL PROPERTIES OF COMMERCIAL SAND-CAST ALUMINUM ALLOYS.

| Alloy | Yield Strength (Set = 0.2 per cent), lb. per sq. in. | Tensile Strength, lb. per sq. in. | Elongation in Four Diameters, per cent | Reduction of Area, per cent | Brinell Hardness Number, 500-kg. load, 10-mm. ball | Shearing Strength, lb. per sq. in. | Endurance Limit, lb. per sq. in. (500,000,000 cycles) | Ratio, Endurance Limit to Tensile Strength |
|---|---|---|---|---|---|---|---|---|
| 12 | 15 700 | 26 150 | 2.0 | 2.8 | 67 | 22 800 | 8 000 | 0.31 |
| 43 | 9 400 | 19 100 | 4.0 | 2.7 | 40 | 15 000 | 6 500 | 0.34 |
| 47 | 11 800 | 27 400 | 9.2 | 10.3 | 50 | 18 800 | 6 000 | 0.22 |
| A108 | 21 000 | 25 900 | 1.8 | 0.0 | 74 | 22 900 | 8 500 | 0.33 |
| 109 | 19 000 | 26 500 | 0.8 | 0.0 | 79 | 22 750 | 10 000 | 0.38 |
| 112 | 20 500 | 25 400 | 0.8 | 0.0 | 79 | 23 700 | 9 000 | 0.35 |
| 121 | ...... | 23 300 | 0.6 | 0.0 | 88 | 23 200 | 10 000 | 0.43 |
| 122 | ...... | 29 600 | 0.7 | 0.0 | 98 | 25 550 | 9 500 | 0.32 |
| 142 | ...... | 29 100 | 0.5 | .... | 96 | 27 100 | 8 000 | 0.28 |
| 142-T61 | ...... | 42 000 | 0.3 | 0.0 | 130 | 35 450 | 8 000 | 0.19 |
| 195-T4 | 22 300 | 32 900 | 5.0 | 4.3 | 74 | 29 050 | 5 500 | 0.17 |
| 195-T6 | 24 000 | 34 100 | 3.0 | 4.7 | 74 | 27 450 | 6 500 | 0.19 |
| 195-T62 | 39 000 | 44 700 | 0.8 | 0.0 | 102 | 33 000 | 7 000 | 0.16 |
| A355-T6 | 28 800 | 35 600 | 1.7 | .... | 91 | 30 200 | 7 000 | 0.20 |
| 356-T6 | 30 200 | 36 400 | 2.0 | .... | 84 | 26 250 | 8 000[a] | 0.22 |
| 406 | 8 100 | 17 700 | 10.3 | 13.9 | 31 | 12 400 | 5 500 | 0.31 |
| 645 | 31 500 | 38 700 | 1.3 | 0.0 | 86 | 29 000 | 8 000 | 0.21 |

[a] Tentative.

NOTE.—The above mechanical properties obtained from A.S.T.M. standard ½-in. diameter tension-test bars cast to size.

TABLE III.—MECHANICAL PROPERTIES OF COMMERCIAL MAGNESIUM ALLOYS.

| Alloy | Size, in. | Form | Yield Strength (Set = 0.2 per cent), lb. per sq. in. | Tensile Strength, lb. per sq. in. | Elongation in Four Diameters, per cent | Reduction of Area, per cent | Brinell Hardness Number, 500-kg. load, 10-mm. ball | Shearing Strength, lb. per sq. in. | Endurance Limit, lb. per sq. in. (500,000,000 cycles) | Ratio, Endurance Limit to Tensile Strength |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | **WROUGHT ALLOYS** | | | | | | |
| 4 per cent aluminum | ¾ | Extruded rod | 18 750 | 35 650 | 19.5 | 28.2 | 46 | .... | 9 000 | 0.25 |
| AM 4.4 | 1¼ | Forged plate | 25 000 | 34 400 | 5.9 | 6.1 | 46 | 18 500 | 10 000 | 0.29 |
| AM 61S | ¾ | Extruded rod | 26 200 | 38 950 | 9.9 | 15.8 | 43 | 17 000 | 13 000 | 0.33 |
| AM 61S | ¾ | Forged plate | 19 700 | 31 050 | 4.8 | 6.8 | 43 | 18 350 | 9 000 | 0.29 |
| AM 61ST | ¾ | Forged plate | 18 500 | 31 700 | 6.2 | 6.0 | 43 | 16 200 | 9 000 | 0.28 |
| | | | | **SAND-CAST ALLOYS** | | | | | | |
| AM 7.4 | ¾ | Test bar | 9 700 | 24 375 | 5.9 | 5.4 | 45 | 17 800 | 6 000 | 0.25 |
| AM 7.4-HT | ¾ | Test bar | 10 125 | 30 700 | 9.2 | 9.0 | 44 | 17 600 | 7 000 | 0.23 |
| AM 240-HT | ½ | Test bar | 13 200 | 35 950 | 11.3 | .... | .. | 19 950 | 10 000 | 0.28 |
| AM 240-HTA-2 | ½ | Test bar | 22 150 | 37 300 | 2.3 | .... | .. | 23 500 | 8 000 | 0.21 |
| AM 764-HTA-1 | ½ | Test bar | 21 600 | 32 750 | 4.5 | .... | .. | 32 750 | 7 000 | 0.21 |

Upon breakage or failure of the specimen, the air pressure is released and in so doing a relay is actuated which stops the machine. This machine can be operated at speeds varying from 600 to 1800 r.p.m.

## FATIGUE TEST RESULTS

The fatigue data from rotating-beam tests (R. R. Moore type) of most of the commercial wrought aluminum alloys, together with their ordinary static mechanical properties, are given in Table I. Similar data for the more common commercial cast alloys are given in Table II. Some data for magnesium and its alloys are given in Table III. The nominal chemical compositions of the aluminum alloys referred to in these tables are given in Table IV.

TABLE IV.—NOMINAL CHEMICAL COMPOSITION OF ALUMINUM ALLOYS.

| Alloy | Copper, per cent | Iron, per cent | Silicon, per cent | Manganese, per cent | Magnesium, per cent | Zinc, per cent | Nickel, per cent | Chromium, per cent |
|---|---|---|---|---|---|---|---|---|
| 2S | .... | .. | .... | .... | .... | .... | .... | .... |
| 3S | .... | .. | .... | 1.25 | .... | .... | .... | .... |
| 4S | .... | ... | .... | 1.25 | 1.0 | .... | .... | .... |
| 43S | .... | ... | 5.0 | .... | .... | .... | .... | .... |
| 14S | 4.4 | ... | 0.8 | 0.75 | 0.35 | .... | .... | .... |
| 17S | 4.0 | ... | .... | 0.5 | 0.5 | .... | .... | .... |
| A17S | 2.5 | ... | .... | .... | 0.3 | .... | .... | .... |
| B17S | 3.5 | ... | .... | .... | 0.3 | .... | .... | .... |
| C17S | 4.0 | ... | 1.25 | 0.5 | 0.5 | .... | .... | .... |
| 24S | 4.2 | ... | .... | 0.6 | 1.5 | .... | .... | .... |
| 25S | 4.4 | ... | 0.8 | 0.8 | .... | .... | .... | .... |
| 32S | 0.8 | ... | 12.5 | .... | 1.15 | .... | 0.8 | .... |
| 51S | .... | ... | 1.0 | .... | 0.6 | .... | .. | .... |
| A51S | .... | ... | 1.0 | .... | 0.6 | .... | .... | 0.25 |
| 53S | .... | ... | 0.7 | .... | 1.25 | .... | .... | 0.25 |
| 12 | 0 | ... | .... | .... | .... | .... | .... | .... |
| 43 | .... | ... | 5.0 | .... | .... | .... | .... | .... |
| 47 | .... | ... | 12.5 | .... | .... | .... | .... | .... |
| A108 | 4.5 | ... | 5.75 | .... | .... | .... | .... | .... |
| 109 | 12.0 | ... | .... | .... | .... | .... | .... | .... |
| 112 | 7.5 | 1.2 | .... | .... | .... | 1.5 | .... | .... |
| 121 | 12.0 | ... | .... | 1.0 | .... | .... | .... | .... |
| 122 | 10.0 | 1.2 | .... | .... | 0.2 | .... | .... | .... |
| 142 | 4.0 | ... | .... | .... | 1.5 | .... | 2.0 | .... |
| 195 | 4.0 | ... | .... | .... | .... | .... | .... | .... |
| A355 | 1.4 | ... | 5.0 | 0.75 | 0.5 | ... | 0.75 | .... |
| 356 | .... | ... | 7.0 | .... | 0.3 | .... | .... | .... |
| 406 | .... | ... | .... | 2.0 | .... | .... | .... | .... |
| 645 | 2.5 | 1.5 | ... | .... | .... | 11.0 | .... | .... |

It is of considerable interest to note the range of endurance ratios (endurance limit to tensile strength) found from the tests. The wide variation in the values for non-ferrous metals has been noted by other investigators.[1] From a study of the data just indicated, however, it would appear that certain additional facts can be pointed out. The higher values of the ratio for the pure metals or those of low alloy content, in the annealed wrought condition (2SO, 3SO, 4SO, 17SO, 25SO, 43SO, 51SO, 53SO, etc.), are analogous to similar values found for low-carbon hot-rolled or annealed steels. In general, the ratio values range from 0.40 to 0.55. In every instance it may be noted that the endurance limit is greater than the yield strength value. In such cases it is often very difficult to obtain satisfactory rotating-beam results because of the plastic flow of the metal. Quite frequently the specimens fail by bending rather than by the usual type of fatigue fracture. Some of the endurance-limit values represent merely the maximum stress at which specimens would run a few hundred million cycles without failure. At slightly higher stresses, the specimens would run for considerable time but eventually fail by bending. Some of the alloys would run longer (and actually fracture) when tested at 3500 r.p.m. than when tested at 1800 r.p.m., indicating that the speed of testing is of some importance when the stresses are in the plastic range.

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[1] J. R. Townsend and C. H. Greenall, "Fatigue Studies of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 353 (1929).

R. R. Moore, "Some Fatigue Tests on Non-Ferrous Metals." *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 66 (1925).

J. B. Johnson and T. T. Oberg, "Fatigue Resistance of Some Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 339 (1929).

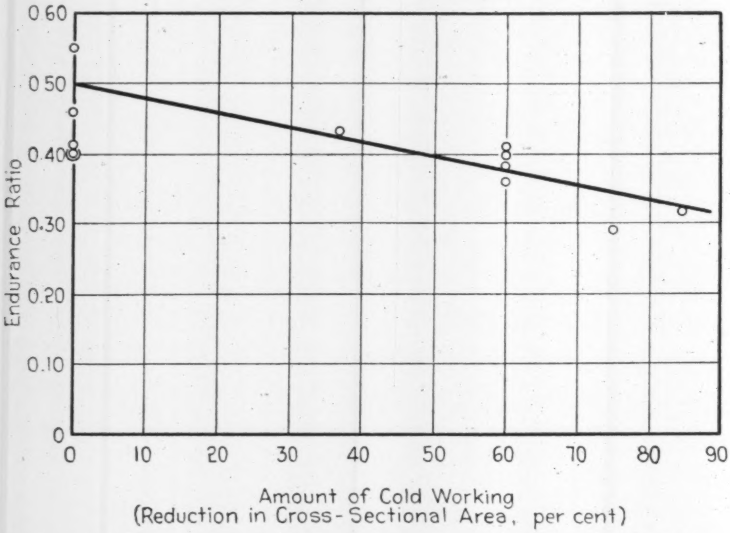

FIG. 8.—Effects of Cold Working on the Endurance Ratio of Aluminum Alloys.

From a study of these observations, it is thought that the endurance ratios of most, if not all, of these alloys, in the properly annealed wrought condition would, under suitable test conditions, be found in the neighborhood of 0.5. Cold working obviously increases the fatigue strength of the metals as can be seen in the case of material

2S, 3S, and 4S. The increase in the fatigue strength, however, is not in direct proportion to the increase in the tensile strength, hence there must be a change in the endurance ratios, and this change seems to be invariably a decrease.

Plotting the endurance ratios as ordinates and the amount of cold working in terms of reduction in cross-sectional area as abscissas, for the cold-worked wrought aluminum alloys, there is obtained the relationship shown in Fig. 8. Reasons for the scatter of the points for the annealed materials have already been suggested. These observations would appear to explain, in part at least, the variations in the endurance ratio values for cold-worked alloys of aluminum. Perhaps a similar consideration of the fatigue data pertinent to other cold-worked metals would explain similar differences.

TABLE V.—EFFECTS OF NOTCHES ON THE FATIGUE STRENGTHS OF ALUMINUM ALLOYS.

| Alloy | Size, in. | Form | Type of Notch | Elongation in 2 in., per cent | Endurance Limit, lb. per sq. in. | | Reduction Caused by Notch, per cent |
|---|---|---|---|---|---|---|---|
| | | | | | Standard | Notched | |
| 17ST....... | ¾ | Extruded and drawn rod............ | 60 deg. sharp V ... | 21.5 | 15 000 | 9 000 | 40 |
| X56SO..... | ½ | Extruded and drawn rod............ | 60 deg. sharp V ... | 26.0 | 19 000 | 6 000 | 68 |
| X56S¾H.... | ½ | Extruded and drawn rod............ | 60 deg. sharp V ... | 10.8 | 21 000 | 2 500 | 88 |
| 4SO........ | ½ | Extruded and drawn rod............ | 60 deg. sharp V ... | 23.0 | 14 000 | 4 000 | 71 |
| 4S¾H...... | ½ | Extruded and drawn rod............ | 60 deg. sharp V ... | 6.5 | 15 000 | 2 500 | 83 |

Heat treatment (of the heat-treatable aluminum alloys) undoubtedly has a beneficial effect on their fatigue strengths as shown in the case of 17SO *versus* 17ST, 25SO *versus* 25SW or 25ST, 51SO *versus* 51SW or 51ST. If consideration is given to alloys of the 51S or 25S type, which are relatively stable at room temperatures in their "as quenched" (W), or solution heat-treatment condition, it may be seen that such heat treatment alone causes most of the beneficial effects on the fatigue strength and little if any benefit is obtained when additional precipitation or final aging treatment is given (*T* condition). As in the case of cold working, where heat treatment causes a marked increase in the tensile strength, there seems to be an effect of lesser amount on the fatigue strength so we must expect a change (decrease) in the endurance ratio. There seems to be some semblance of agreement in the endurance-ratio values for the commercial heat-treated wrought alloys of aluminum since they nearly all lie between 0.2 and 0.3, averaging about 0.25.

Gillett[1] has pointed out the susceptibility of hard steels to notch effect. He stated that for equal hardness the tougher and more ductile the steel and the higher its notch-toughness, the better. On the other hand, Moore[2] failed to find any satisfactory correlation between ductility as measured by elongation and the notch-fatigue strength of metals. The data given in Table V would indicate that as the elongation is reduced by cold working, notches become much more deleterious in their effects on the fatigue strengths of aluminum alloys. The effect of notches (60 deg. sharp V) on the heat-treated aluminum alloys having considerable elongation is typified by the data for 17ST given in the table. When such heat-treated alloys have appreciably lower elongations, greater deleterious effects would be expected. On the other hand, the numerical values of the elongation *per se* cannot be used as a satisfactory basis for predicting the effect of notches on the fatigue strengths of the various alloys in all their different conditions. In so far as a comparison can be made, these data show rather satisfactory agreement with similar data given by Moore.[2] He found that a 60-deg. V notch with 0.10 in. radius lowered the fatigue limit of hard-rolled commercially pure aluminum (2SH) 63 per cent and of a heat-treated alloy of the duralumin type (25ST) 45 per cent.

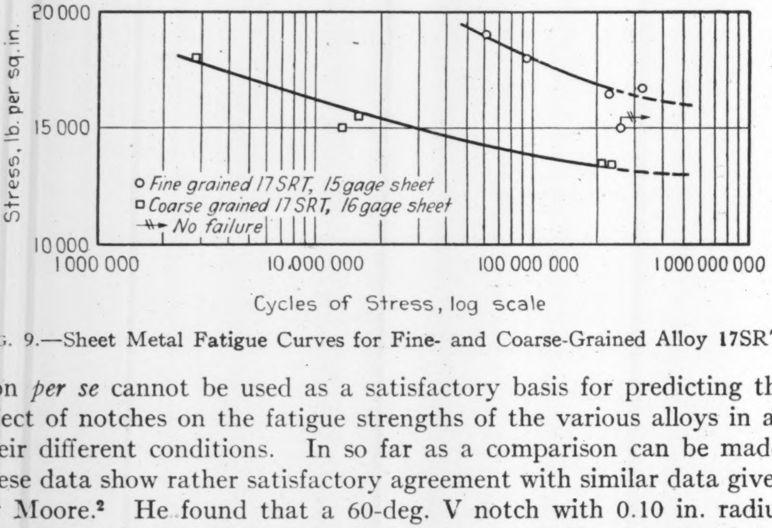

FIG. 9.—Sheet Metal Fatigue Curves for Fine- and Coarse-Grained Alloy 17SRT.

Grain size in a metal undoubtedly affects its endurance limit but to what extent quantitatively is often a matter of conjecture. The curves shown in Fig. 9 show the relatively slight lowering of the fatigue strength of 17SRT sheet when excessively large grain size obtains. In the one lot of material there were less than one-fifteenth as many grains per square millimeter as in the other, yet the decrease in fatigue strength caused was only 3000 lb. per sq. in. or about 19 per cent. Similar effects have been obtained in large forgings having large grain structure.

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[1] Report of Research Committee on Fatigue of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 259 (1930).

[2] R. R. Moore, "Some Fatigue Tests on Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 66 (1925).

Comparison of fatigue data from different lots of the same nominal material in different fabricated forms and involving different types of fatigue tests show but relatively small variations in the fatigue limits obtained. This is exemplified by results on 17ST sheet, extruded rod, rolled rod and tubing; the value found for the endurance limit being 15,000 lb. per sq. in. ± 1000 lb. per sq. in. on the basis of 500,000,000 cycles of stress. Comparison of many fatigue data on quite similar materials, obtained at such laboratories as the U. S. Bureau of Standards, the Wright Field laboratories of the U. S. Air Service, the Engineering Experiment Station of the U. S. Navy at Annapolis and the Aluminum Research Laboratories have invariably shown quite satisfactory agreement, the order of difference being generally within the limits just indicated.

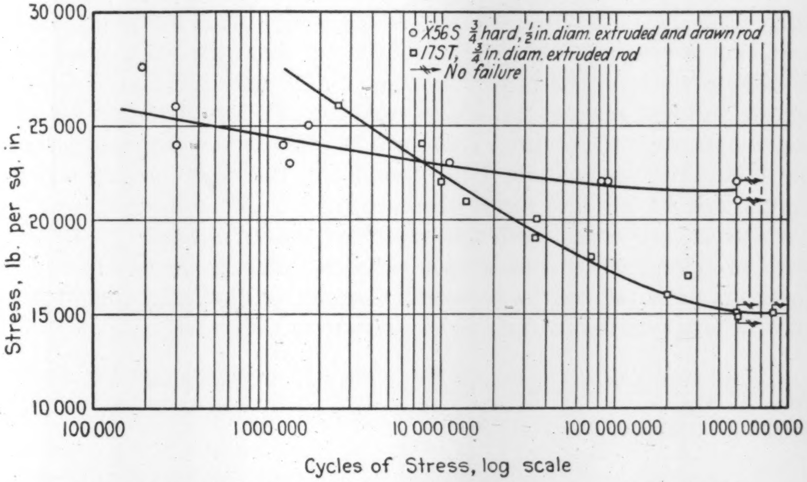

FIG. 10.—Rotating-Beam Fatigue Curves for Aluminum-Alloy Rod.

Comparison of the same fatigue data with foreign laboratory test results on similar materials, however, frequently reveals marked differences. The main cause for such differences appears to be in the number of cycles of stress used as a basis for determining the endurance limit. Many foreign investigators apparently have been accustomed to stop their fatigue tests after 20 to 50 million cycles, whereas the majority of those testing light alloys in this country have not been satisfied with results based on less than 300 to 500 million cycles. More recently there seems to be justification for considering results based on much longer tests. Certainly tests on the basis of 1 to 10 million cycles can give very misleading results in the case of some light alloys as shown by the two sets of data plotted in Fig. 10. Here the really marked difference in fatigue strength of the two materials is not revealed until the tests have been run 400 or 500 million cycles, then the alloy which appeared to be the superior at first becomes decidedly the inferior in fatigue strength.

Reference to the mechanical properties shown for the cast aluminum alloys in Table II reveals the fact that the endurance ratios vary from 0.16 to 0.43. Comparisons of these values with those indicated for both wrought magnesium and cast magnesium, given in Table III, shows that the values for the magnesium alloys in either condition are appreciably more consistent than the values for the aluminum alloys in the same conditions. The endurance-ratio values for the wrought magnesium alloys vary from 0.25 to 0.33 and for the cast magnesium alloys from 0.21 to 0.28. So far no high values for the endurance ratio have been found for the light casting alloys comparable to the high ratios obtained by Kommers[1] on cast iron.

For a very complete bibliography of results obtained in fatigue tests of light metals and alloys, reference should be made to the abstracts prepared by the Research Committee on Fatigue of Metals and those appearing in the abstracts of *Metals and Alloys*.

*Acknowledgment.*—The author wishes to acknowledge the assistance of Messrs. F. M. Howell and M. F. Rupp in carrying out the tests and preparing the data.

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[1] J. B. Kommers, "The Effect of Under-Stressing on Cast Iron and Open-Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

# DISCUSSION

MR. L. B. TUCKERMAN[1] (*presented in written form*).—Mr. Templin has again added materially to our knowledge of the properties of light metals and alloys. The knowledge of fatigue properties of metals is ever becoming more important and a summary of test methods and test results such as this is of great value. We need to know much more about the behavior of materials under varying stresses where the average stress is not zero, and Mr. Templin with his new machine for direct compression and tension and his apparatus for combined tension and bending is helping to fill that gap.

Everyone interested in fatigue testing should study carefully his Fig. 10 and the paragraph in which it is discussed. It shows more clearly than any series of tests that I have previously seen how unreliable are conclusions concerning the fatigue strength of nonferrous alloys drawn from tests carried only to 10 or 20 million cycles.

MR. J. A. GANN[2] (*presented in written form*).—Our experience in the fatigue endurance of light alloys has been primarily with a series of magnesium-base alloys. Some of the data we have obtained are submitted here as they supplement the data presented by Mr. Templin.

Table I gives the nominal composition and the properties in the cast and wrought conditions of the alloys under consideration. Although Dowmetal M is seldom used in the cast form, its properties have been included in order to illustrate more fully the effect of alloy composition. All fatigue endurance values are based on 500 million cycles. The ranges in properties are those normally encountered in regular production work. The ratios of fatigue endurance to tensile strength, calculated from the minimum property values of Table I, agree so closely with those calculated from the maximum property values of the same table that a single value is an adequate expression of this ratio. So far as comparisons are possible, these data are in fairly good agreement with those presented by Mr. Templin.

Ratios of fatigue endurance to yield strength and to percentage elongation have been calculated. They are not included as they are not so consistent as the fatigue endurance to tensile strength ratios and fail to bring new relationships.

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[1] Assistant Chief, Division of Mechanics and Sound, U. S. Bureau of Standards, Washington, D. C.

[2] Chief Metallurgist, The Dow Chemical Co., Midland, Mich.

TABLE I.—MECHANICAL PROPERTIES OF COMMERCIAL MAGNESIUM ALLOYS.

| | Nominal Composition, per cent | Form | Tensile Strength, lb. per sq. in. | Yield Strength (Set=0.2 per cent), lb. per sq. in. | Elongation in Four Diameters, per cent | Endurance Limit, 500,000,000 Cycles, lb. per sq. in. | Ratio, Endurance Limit to Tensile Strength |
|---|---|---|---|---|---|---|---|
| | | | SAND CAST ALLOYS | | | | |
| Dowmetal M | 1.5 Manganese | Heat Treated | 13 000 to 21 000 | 5 000 to 8 000 | 3 to 8 | 3 000 to 4 000 | 0.21 |
| Dowmetal E | 6.0 Aluminum<br>0.3 Manganese | As Cast | 26 000 to 32 000 | 8 000 to 11 000 | 7 to 14 | 6 000 to 7 000 | 0.22 |
| Dowmetal A | 8.0 Aluminum<br>0.2 Manganese | As Cast | 23 000 to 28 000 | 10 000 to 14 000 | 3 to 7 | 6 000 to 8 000 | 0.28 |
| | | Heat Treatment No. 1 | 30 000 to 37 000 | 10 000 to 13 000 | 7 to 13 | 6 000 to 8 000 | 0.21 |
| | | Heat Treatment No. 2 | 30 000 to 37 000 | 10 000 to 14 000 | 6 to 12 | 6 000 to 8 000 | 0.21 |
| | | Heat Treatment No. 3 | 30 000 to 38 000 | 12 000 to 16 000 | 4 to 9 | 6 000 to 8 000 | 0.21 |
| Dowmetal G | 10.0 Aluminum<br>0.1 Manganese | As Cast | 20 000 to 25 000 | 12 000 to 16 000 | 1 to 4 | 8 000 to 10 000 | 0.40 |
| | | Heat Treatment No. 1 | 29 000 to 37 000 | 10 000 to 15 000 | 5 to 10 | 9 000 to 11 000 | 0.30 |
| | | Heat Treatment No. 2 | 29 000 to 38 000 | 14 000 to 20 000 | 2 to 7 | 8 000 to 10 000 | 0.27 |
| | | Heat Treatment No. 3 | 29 000 to 38 000 | 17 000 to 22 000 | 1 to 4 | 8 000 to 10 000 | 0.27 |
| | | | WROUGHT ALLOYS | | | | |
| Dowmetal M | 1.5 Manganese | Extruded | 37 000 to 45 000 | 23 000 to 29 000 | 4 to 12 | 5 000 to 8 000 | 0.16 |
| Dowmetal F | 4.0 Aluminum<br>0.3 Manganese | Extruded | 37 000 to 43 000 | 24 000 to 33 000 | 12 to 18 | 13 000 to 16 000 | 0.36 |
| | | Forged | 34 000 to 42 000 | 19 000 to 29 000 | 6 to 15 | 11 000 to 13 000 | 0.32 |
| Dowmetal E | 6.0 Aluminum<br>0.3 Manganese | Extruded | 40 000 to 45 000 | 26 000 to 33 000 | 10 to 17 | 15 000 to 17 000 | 0.38 |
| | | Forged | 34 000 to 43 000 | 16 000 to 28 000 | 6 to 17 | 11 000 to 13 000 | 0.31 |
| Dowmetal A | 8.0 Aluminum<br>0.2 Manganese | Extruded | 44 000 to 50 000 | 29 000 to 35 000 | 9 to 16 | 16 000 to 19 000 | 0.37 |

The following conclusions can be drawn:

1. The fatigue endurance limit of magnesium-base alloys of the magnesium-aluminum-manganese series increases with the percentage of aluminum in both the cast and wrought alloys.

2. The ratio of fatigue endurance to tensile strength for cast alloys in the magnesium-aluminum-manganese series varies from 0.20 to 0.40, averaging approximately 0.25.

3. The ratio of fatigue endurance to tensile strength for wrought alloys in the magnesium-aluminum-manganese series (excepting alloy M) varies from 0.31 to 0.38, averaging approximately 0.35.

4. The ratio of fatigue endurance to tensile strength in the magnesium-aluminum-manganese series of alloys tends to increase with the percentage of aluminum in the alloy.

5. Heat treatment very materially increases the tensile strength of cast magnesium-aluminum-manganese alloys containing from 8 to 12 per cent aluminum, but has little to no effect on the fatigue endurance. As a result, the ratio of fatigue endurance to tensile strength is highest in the cast condition. A similar effect was observed by Mr. Templin on aluminum alloy No. 142.

6. Cast magnesium alloys and cast aluminum alloys have the same general range of fatigue endurance limit values.

7. Wrought magnesium alloys and wrought aluminum alloys have the same general range of fatigue endurance limit values.

8. Weight for weight, magnesium alloys have higher fatigue endurance limit values than aluminum alloys.

In conclusion we wish to ask Mr. Templin what observations he has made regarding the effect of specimen diameter on the fatigue endurance limit. Estimated values from uncompleted tests on one lot of coarse-grained extruded Dowmetal M are:

| SPECIMEN DIAMETER, IN. | FATIGUE ENDURANCE, LB. PER SQ. IN. |
|---|---|
| 0.30 | 5000 |
| 0.35 | 7000 |
| 0.40 | 8500 |

Specimen diameter, however, appears to have little or no effect on the fatigue endurance of finer grained alloys. In view of these findings and Mr. Templin's observations that the fatigue endurance increases with decreasing grain size, we venture to generalize that fatigue endurance is dependent on specimen diameter in the case of relatively coarse-grained metals only.

**MR. A. V. DE FOREST.**[1]—Mr. Templin has machinery that will test greater lengths of material than, so far as I know, anybody else has attempted to handle. If the results show that the lowest strength in 50 ft. is close to the usual results obtained on the usual length of a few tenths of an inch, we would have much more confidence in the results of fatigue tests on normal test specimens. A confirmation of that fact would be of assistance to many people who are dependent on fatigue tests on very minor volumes of material, whereas in actual practice the stresses are borne by very large volumes of material.

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[1] Consulting Engineer, American Chain Co., Inc., Bridgeport, Conn.

MR. H. J. GOUGH.[1]—Reference is made in Mr. Templin's paper to "foreigners" which is somewhat misleading, as the impression is given that the importance of fatigue tests on certain alloys conducted on a very lengthy endurance basis is insufficiently appreciated in other countries.

In my own laboratory we always report a fatigue result as an endurance limit on a stated number of reversals, under stated conditions of stress, environment, temperature, etc., and the figure quoted relates simply to those test conditions. Mr. Templin may carry his test on to more extended endurances, obtaining another figure; I do not think, however, that Mr. Templin would be quite sure that, with certain materials, if he carried his tests on to even greater endurances, he would always get the same endurance limits as those obtained from 500 million reversals.

The point is that, unless an $S$-$N$ diagram has become definitely asymptotic to some stress value, the result of the test should always be given as an endurance limit on a stated reversals basis: in general, there is no magic number of reversals associated with a definite limiting range of stress.

But I do not wish to be misunderstood: these lengthy fatigue tests of Mr. Templin and his associates are greatly welcomed and appreciated by all interested in fatigue phenomena.

A matter arises in which I am greatly interested. Even assuming one has the limiting fatigue range of a material determined under simple stressing actions (direct, flexural or torsional), how are these results to be applied in the design of a component which is subjected to a complex stress distribution? It is surprising to find how neglected has been the field of the resistance of materials to *combined* fatigue stresses. Is there some general stress criterion governing fatigue failure? That is the problem and an investigation into combined stresses offers the direct method of attack. There seems little doubt that one reason for the gap in our knowledge is associated with the difficulty of devising satisfactory high-speed testing machines for applying, simultaneously, and in phase, cycles of at least two distinct types of stressing action. A fairly simple type of machine has been designed and constructed recently at the National Physical Laboratory. Working at a speed of 2000 cycles per minute, the whole range of combined reversed torsion and reversed bending—from simple torsion to simple plane bending—can be investigated. The results of the first series of tests have been referred to in the eighth Marburg Lecture. The behavior of this one material, mild steel, was in extraordinarily good agreement with the relation of V. Mises. This must not yet, of course, be taken as the *general* criterion for fatigue failure: a comprehensive series of tests is now in hand on a variety of materials. Should it emerge that a general law of failure applies to metals, an important step will be made towards the simplification of the subject of fatigue, but it may be that no such general law obtains. In any case, experimental data are urgently required and I can recommend the field of combined fatigue stresses as one well worthy of the attention of several independent investigators or laboratories.

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[1] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

MR. J. R. TOWNSEND.[1]—I should like to ask Mr. Templin whether there is any change in the endurance tensile strength ratio on heat-treated duralumin that has been rolled after heat treatment? Were the specimens tested transversely or longitudinally with regard to the direction of rolling? Is there an optimum grain size with regard to the endurance limit of duralumin? I should also like to ask whether Mr. Templin has any information concerning the nature of the fatigue cracks in fine-grained and also in coarse-grained aluminum alloys?

MR. R. L. TEMPLIN.[2]—We have not noticed any difference in the fatigue cracks as suggested by Mr. Townsend. I do not know that we have gone into it far enough to say there is no difference, but we have not noticed any. For the duralumin rolled after heat treatment, tested both longitudinally and transversely, we do sometimes find a slight difference, and I believe it is in favor of the transverse specimens. I refer to materials such as are used in lighter-than-air craft.

Referring to Dr. Gough's comments I certainly meant no offense to investigators in the foreign field in the fatigue of metals; I simply wanted to emphasize in this paper that the basis used in determining or stating the fatigue limits is frequently quite different abroad than it is in this country, and where that really worries the commercial producer of course is that the prospective user of the product says, "Why can't you produce metal that has a fatigue limit some thousand pounds per square inch higher, as they do abroad?" Apparently the questioner does not know anything about the basis used in defining that value. An interesting point came up at the winter meeting of the British Institute of Metals in the proposal of another short-time fatigue test involving duralumin. The author in that case carried out his tests to some 25 million cycles and on that basis found a discrepancy between the so-called long-time and short-time test of some eleven per cent. Plotting his data on our duralumin fatigue curve showed it checked beautifully as far as he went, about 25 million cycles, but on the basis of 500 million cycles the discrepancy between the long and short-time fatigue test was in the neighborhood of thirty-three per cent.

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[1] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

[2] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

Answering Mr. Gann's question—we found no differences in the fatigue values in so far as our tests have gone, dependent on the size of specimen. In the case of fine-grained material we do not have tests that exactly parallel his. When it comes to coarse-grained material, the results are about what we would expect, assuming rather large crystal size. That is, where the crystal size is on the same order of magnitude as the diameter of the test specimen, we would naturally expect a definite effect, and as the specimen size is increased while the crystal size remains constant, there would appear to be a change in the fatigue properties.

Mr. Townsend asked whether there was an optimum grain size. I do not know whether there is an optimum one or not. In general, the finer the grain the better the values that will be obtained. So far, I know of no data that would indicate there is a critical point beyond which the fatigue value would again decrease.

# CONSISTENT DATA SHOWING THE INFLUENCE OF WATER VELOCITY AND TIME ON THE CORROSION OF IRON

By R. F. Passano[1] and F. R. Nagley[1]

## Synopsis

With temperature, oxygen concentration, composition of water, area of specimen, surface condition of specimen and composition of metal controlled, the influence of water velocity and time were studied in an immersion test, which is described. Successive experiments were conducted at intervals between 1 and 60 days at a velocity of 1 meter per minute, between 1 and 30 days at 2 meters per minute, and between 1 and 15 days at 5 meters per minute. It is shown that the losses from successive experiments under the same essential conditions did not differ by more than should be left to chance. The curves showing the relation between time and average loss of weight were drawn so they did not miss the experimental values by more than the errors of the averages. Iron loses weight in direct proportion to time at the beginning of the tests. The duration of this initial period decreases as the velocity is raised, and is probably a hyperbolic function of the velocity. Following the initial period, iron loses weight in proportion to the logarithm of time under the conditions of these tests. From the relative position of the logarithmic portions of the loss-time curves, one would infer that the curves representing the various velocities will cross. Thus, the loss of weight from iron specimens increases with velocity for a time, but in the end the specimens exposed at high velocities will probably lose less weight than those exposed at low velocities.

Last year one of the authors presented a paper before the Society[2] which showed data from an immersion test on iron under very specific conditions of test which were maintained while the experiments were in progress. These data represented the loss of weight from test areas of 20 sq. cm. exposed to Central Works tap water[3] which was made to flow by the specimens at 1 meter per minute. The experiments covered continuous tests of 3, 6, 9, 12, and 15 days duration. In order to be sure that the average loss of weight reported for these various time intervals was not accidental, successive experiments were conducted for each of these intervals and it was shown that the losses in successive experiments under the same essential conditions did not differ by more than should be left to chance.

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[1] Research Laboratories, The American Rolling Mill Co., Middletown, Ohio.

[2] R. F. Passano, "Controlled Data from an Immersion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 468 (1932).

[3] Tap water at the Central Works of The American Rolling Mill Co., Middletown, Ohio.

These experiments have been continued and now cover intervals between 1 and 60 days at 1 meter per minute, between 1 and 30 days at 2 meters per minute, and between 1 and 15 days at 5 meters per minute. The relationship between the losses at two time intervals or two velocities was not under consideration at the time the observations were being made. The authors' aim has been to get data which they consider valuable, and then present these data so that the essential information in the original observations is available in the presentation.

Practically it is impossible to measure anything and get the same identical value for individual observations time after time. In other words, the result of a series of measurements is a distribution of values. If one is to convey the information contained in the observations, it is necessary to describe the observed distribution in some way. The recommendations of the Society's Committee on Interpretation and Presentation of Data are given this year in a Manual on Presentation of Data.[1] The data in this paper are summarized in accordance with this committee's recommendations and the essential information in the original observations is given by the observed average, the observed standard deviation, and the number of observations.

## TEST SPECIMENS

The test specimens used in this investigation were prepared from ½-in. round iron bars, of which the following is an analysis:

| | |
|---|---|
| Carbon, per cent | 0.015 |
| Manganese, per cent | 0.016 |
| Phosphorus, per cent | 0.006 |
| Sulfur, per cent | 0.029 |
| Silicon, per cent | 0.002 |
| Copper, per cent | 0.064 |

Each specimen had a dowel pin on its back end by means of which it was held in a specimen holder. Above the dowel pin a cylindrical area of 20 sq. cm. was marked off; this was the test area. The front end of the specimen was stream-lined so the water would pass by it to the test area without turbulence.

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I (1933); also published in pamphlet form.

## SPECIMEN HOLDERS

The top of the specimen holder was a hard-rubber cap with a hole in the center of the top into which the dowel pin of the specimen fitted snugly. If the fit was good, it was not necessary to apply a protective coating to the dowel pin and the back end of the specimen. This hard-rubber cap was firmly attached to a stainless-steel tube perforated near the bottom, and the whole was held concentrically in a glass tube $1\frac{1}{16}$ in. in inside diameter by stainless-steel centering pegs accurately fitted in the hard-rubber cap and a rubber stopper at the bottom through which the stainless-steel tube passed. The end of the stainless-steel tube which projected beyond the rubber stopper was fitted with an adjustable orifice which permitted the flow to be regulated so that the water passed through the ring made

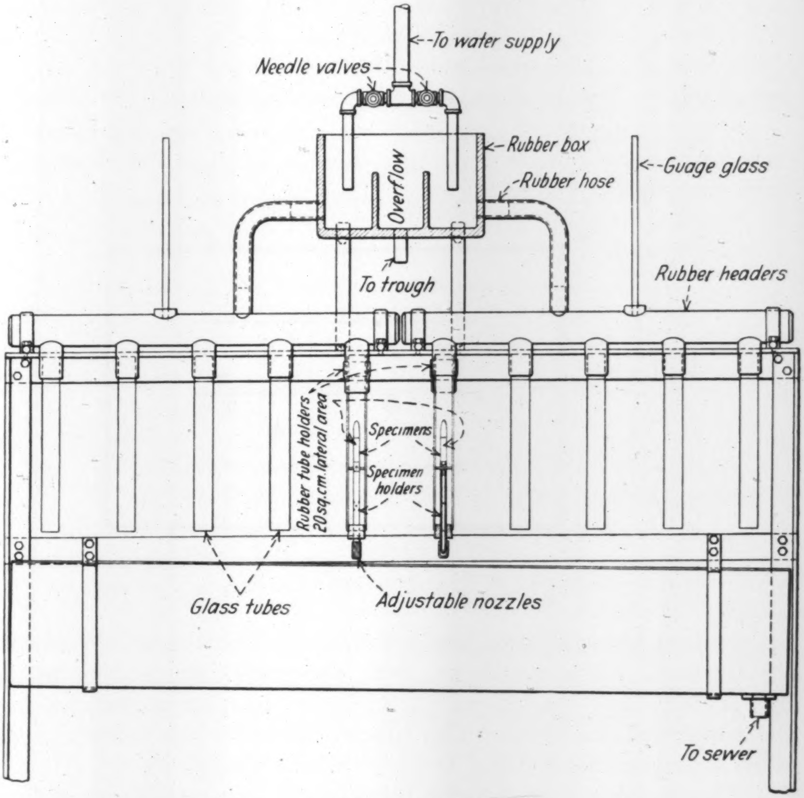
To water supply
Needle valves
Rubber box
Guage glass
Rubber hose
Overflow
To trough
Rubber headers
Rubber tube holders 20 sq.cm. lateral area
Specimens
Specimen holders
Glass tubes
Adjustable nozzles
To sewer

FIG. 1.—Corrosion Testing Apparatus.

by the inside surface of the glass tube and the surface of the test specimen at the desired velocity.[1]

## EXPOSURE AND TEST CONDITIONS

The specimen was cleaned for test and weighed. The nose was then coated with paraffin, and after it had hardened, the specimen was placed in a holder, inserted in a glass tube and the water introduced from the constant-level box to the distributing header, and so to the glass tube containing the specimen. After passing out of the orifice at the bottom of each tube, the water went to the drain; it was not recirculated.[2] The arrangement is shown diagrammatically in Fig. 1.

Five specimens as described were exposed simultaneously in each experiment. After a definite interval of time, the five specimens were removed from the test. The paraffin covering the streamlined end of the specimens was taken off with hot water. Following this operation, the specimens were immersed immediately in 1:1 hydrochloric acid (containing an inhibitor) to remove the rust from the surface of the piece. The specimens were then washed in distilled water and alcohol and finally dried for reweighing.

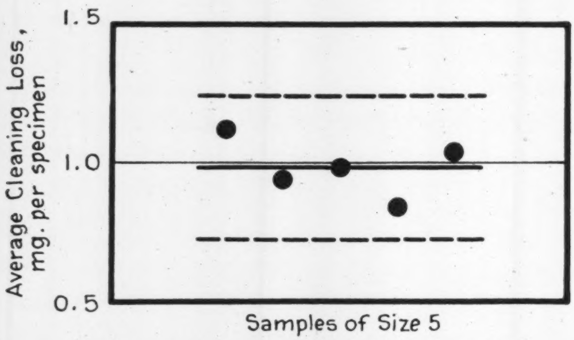

FIG. 2.—Magnitude of Error Introduced by Cleaning Operations.

These cleaning operations introduce a constant error and the magnitude of this effect is shown graphically in Fig. 2. This constant error should be subtracted from all of the values shown in Table III, which values have not been corrected for the reason that the uncertainty of the average value due to the magnitude of the standard deviation and the number of observations is greater in the majority of cases than the constant error of cleaning. In reality, the assurance that one has in an average value depends so much more upon knowledge that the conditions under which the values were taken were controlled than it does on the constant error and the error of the

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[1] The measured velocity is actually the average velocity over the section carrying water. It is known that the velocity near the surface of the specimen and near the surface of the glass tube is less than in the middle of the section due to friction. See any standard text on hydraulics.

[2] Many intangible difficulties can develop when the water is used over and over again.

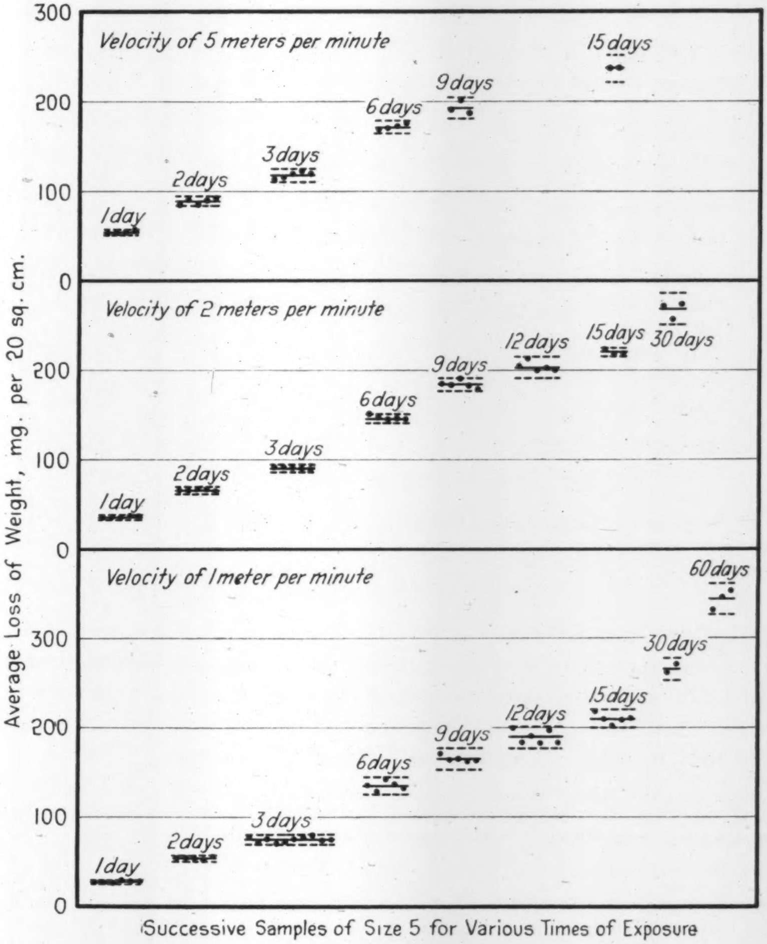

FIG. 3.—Average Loss in Weight of Samples at Various Velocities and Times of Exposures.

average, that the most pertinent bit of evidence that one can produce is evidence that successive experiments made under the same essential conditions do not differ by more than should be left to chance. Such evidence is presented in Fig. 3. It will be noted that in every case at least two experiments, each involving five specimens, have been conducted under the same essential conditions. Control is indicated by the fact that the individual averages lie within limits.[1]

TABLE I.—COMPOSITION OF CENTRAL WORKS TAP WATER.

| | COMPOSITION, P.P.M. |
|---|---|
| Total solids | 522.40 |
| Organic matter | 8.00 |
| Silica ($SiO_2$) | 10.00 |
| Aluminum (Al) | 1.27 |
| Iron (Fe) | 1.20 |
| Calcium (Ca) | 110.54 |
| Magnesium (Mg) | 38.13 |
| Sodium and potassium (Na and K), by difference | 28.74 |
| Chloride (Cl) | 16.00 |
| Sulfate ($SO_4$) | 90.12 |
| Nitrate ($NO_3$) | none |
| Carbonate ($CO_3$) | none |
| Bicarbonate ($HCO_3$) | 218.40 |
| Hydroxide (OH) | none |
| Alkalinity (as $CaCO_3$) | 334.00 |
| Dissolved oxygen[a] | 10.2 |
| pH value | 7.5 |
| Average temperature, deg. Cent.[b] | 14.9 |

[a] Maximum 10.6 p.p.m., minimum 9.9 p.p.m.

[b] For months on end the temperature ranges between 14.5 and 15.5 C.

### *Composition of Water:*

The water going into the constant-level box came direct from the tap. The authors have found that the total solids in this water, its temperature and oxygen concentration do not change appreciably from time to time. The data in Table I are typical.

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[1] See Table I of "Controlled Data from an Immersion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 471 (1932), and the following data from three experiments representing 60 days of continuous exposure to Central Works tap water at a velocity of 1 meter per minute:

| EXPERIMENT | COMPLETED | $\overline{X}$ | $\sigma$ |
|---|---|---|---|
| No. 1–60–1 | 6–22–32 | 352.30 | 12.643 |
| No. 1–60–2 | 2–25–33 | 345.28 | 10.428 |
| No. 1–60–3 | 3– 8–33 | 330.58 | 11.067 |
| | Estimated $\overline{X}'$ = | 342.72 | $\overline{\sigma} = 11.379$ |

$$\pm 3\sigma_{\overline{X}} = \frac{3\overline{\sigma}}{c_2\sqrt{n}} = 18.17 \begin{cases} 360.89 \\ 324.55 \end{cases}$$

The three averages above should lie within the limits shown, 324.55 and 360.89, if the variation is no greater than should be left to chance. The symbol $\overline{X}'$ is rightly used only when it indicates the true average of the universe. Thus the average of the losses from the three experiments above is only an estimate of the true average of the universe and is rightly referred to elsewhere in the text as $\overline{X}$ rather than $\overline{X}'$.

*Operation and Effect of Irregularities:*

Inasmuch as it is not practical to have a man observing the test conditions 24 hours a day, a recording pressure gage was connected to the water line to indicate any irregularity in the amount of water supplied to the constant-level boxes. The authors know from experience the minimum water pressure which is required to keep the apparatus full. If the apparatus is designed so that the rusting specimens are exposed to air instead of water during a temporary shut-down, the loss from the specimens so treated is higher than is the loss from those which are continually exposed to the desired conditions.[1] Similarly, if the apparatus is designed so that the rusting specimens are exposed to stagnant instead of running water during a temporary shut-down, the loss from these specimens is less than from those which are continually exposed to the desired conditions. Thus, it has been our policy to reject from consideration the data on all samples in test at the time of irregular water supply; quite often these samples are discarded without determining the actual loss of weight which they experienced to avoid possible confusion in the records.

TABLE II.—APPEARANCE OF SPECIMENS SHORTLY AFTER EXPOSURE.

| TIME OF OBSERVATION | VELOCITY OF 1 METER PER MINUTE | VELOCITY OF 2 METERS PER MINUTE | VELOCITY OF 5 METERS PER MINUTE |
|---|---|---|---|
| 5 min. | dull gray | metallic luster fading to dull gray | dull gray all over |
| 10 min. | dull gray | blue-green at top | dull gray turning green |
| 20 min. | blue-green at top | blue-green all over | green with small amount of yellow at top |
| 40 min. | same with more blue-green | green with small amount of yellow at top | dark green or black |
| 1 hr. | blue-green all over with some yellow at top | same, but more yellow | same, but darker |
| 6 hr. | Same with more yellow | brown all over | dark brown all over |
| 1 day | brown (yellow or red over black and green) | brown (yellow or red over black) | yellow or brown all over |

The loss of weight from a specimen which has not been attacked all over is low. It will be seen that it is possible to design and operate the immersion testing equipment in such a way that any accident or irregularity will cause low values. Under these circumstances if any values are in error, they will be the low ones.

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[1] This is the apparatus illustrated in Fig. 1.

TABLE III.—SUMMARY AND CONDENSATION OF ESSENTIAL INFORMATION.

| TIME OF CONTINUOUS IMMERSION, DAYS | NUMBER OF OBSERVATIONS[a] | AVERAGE LOSS $\overline{X}$, MG. PER 20 SQ. CM. | AVERAGE STANDARD DEVIATION $\overline{\sigma}$ FOR SAMPLES OF 5 SPECIMENS | ESTIMATED STANDARD DEVIATION $\sigma'$ OF UNIVERSE[b] | $3\sigma$ ERROR OF AVERAGE[c] |
|---|---|---|---|---|---|
| VELOCITY OF 1 METER PER MINUTE | | | | | |
| 1 | 30 | 27.8 | 1.0 | 1.2 | 0.6 |
| 2 | 25 | 54.2 | 2.2 | 2.6 | 1.6 |
| 3 | 50 | 74.9 | 3.6 | 4.3 | 1.8 |
| 6 | 25 | 134.9 | 5.8 | 6.9 | 4.1 |
| 9 | 25 | 165.0 | 7.2 | 8.6 | 5.2 |
| 12 | 30 | 189.4 | 7.9 | 9.3 | 5.1 |
| 15 | 25 | 209.3 | 6.7 | 7.9 | 4.7 |
| 30 | 10 | 265.1 | 7.5 | 9.0 | 8.5 |
| 60 | 15 | 342.7 | 11.4 | 13.6 | 10.5 |
| VELOCITY OF 2 METERS PER MINUTE | | | | | |
| 1 | 25 | 36.2 | 1.3 | 1.5 | 0.9 |
| 2 | 25 | 66.0 | 2.2 | 2.6 | 1.6 |
| 3 | 25 | 90.9 | 2.2 | 2.6 | 1.5 |
| 6 | 25 | 146.4 | 3.1 | 3.7 | 2.2 |
| 9 | 25 | 184.3 | 4.8 | 5.8 | 3.5 |
| 12 | 25 | 203.5 | 7.5 | 8.9 | 5.3 |
| 15 | 15 | 219.6 | 2.9 | 3.4 | 2.6 |
| 30 | 15 | 267.6 | 11.5 | 13.6 | 10.5 |
| VELOCITY OF 5 METERS PER MINUTE | | | | | |
| 1 | 20 | 54.8 | 2.0 | 2.4 | 1.6 |
| 2 | 25 | 89.2 | 2.9 | 3.4 | 2.0 |
| 3 | 25 | 117.7 | 4.3 | 5.1 | 3.1 |
| 6 | 20 | 171.2 | 4.3 | 5.1 | 3.4 |
| 9 | 15 | 192.8 | 7.0 | 8.3 | 6.4 |
| 15 | 10 | 235.5 | 10.0 | 11.9 | 11.3 |

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[a] $m$ samples of size 5; that is, 30 observations signify 6 samples of 5 specimens each.

[b] Taking $\frac{\overline{\sigma}}{c_2}$ as the estimate of $\sigma'$, $c_2 = 0.84069$ for samples of size 5.

[c] Error of average $= \frac{s\sigma'}{\sqrt{n}}$, $s = 3.0$ when probability $P = 0.9973$.

*Preparation of Surface for Test:*

There has been considerable discussion from time to time about the method of preparing the surface of specimens for test. One hesitates rightly to make a definite recommendation on this point,

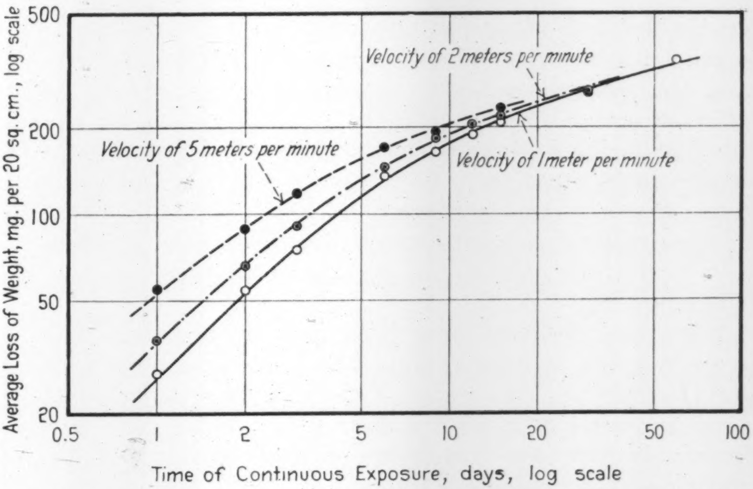

FIG. 4.—Relation of Average Loss to Time of Exposure (log-log coordinates).

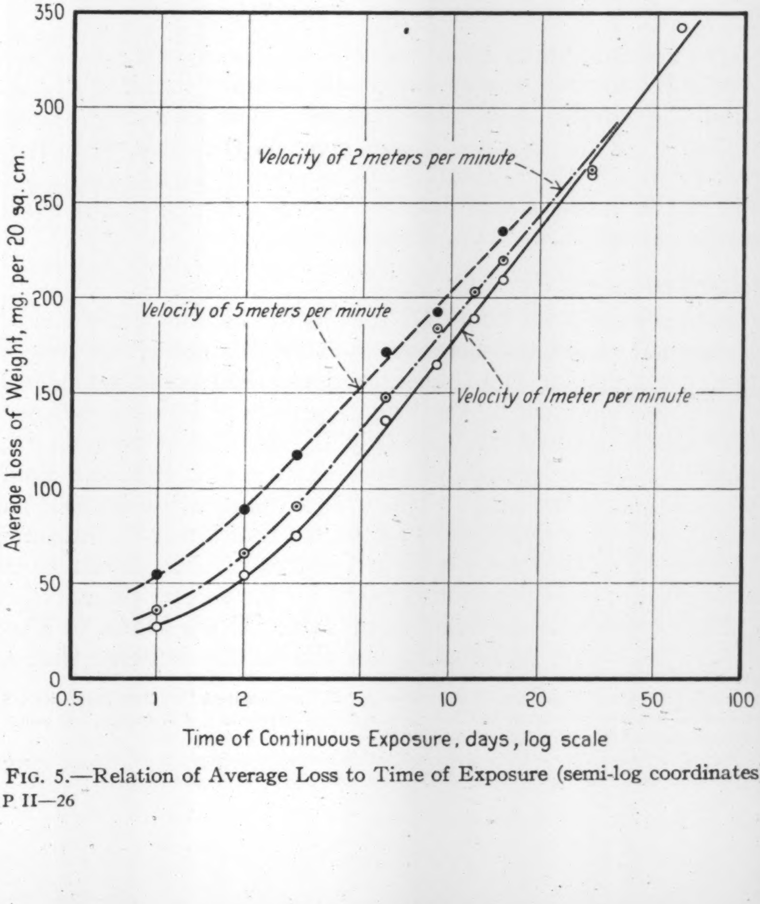

FIG. 5.—Relation of Average Loss to Time of Exposure (semi-log coordinates).

since the method of preparation must be accommodated to the material being tested. We are of opinion that the method of preparing the surface for test is unimportant *provided the specimen corrodes all over.* In the course of our investigations we have used at least six methods of preparing the surface and find that the losses from specimens prepared in these various ways are not significantly different *when the specimens corrode all over.* Some of these methods have been mechanical and some of them have been chemical. We believe that surface difficulties are associated principally with air-formed oxide films.[1]

When the specimens have been properly prepared, they begin to corrode almost as soon as the water comes in contact with them. Table II covers the appearance of the rust for a period of 24 hours at intervals; after 24 hours it is difficult to describe qualitatively the differences between the rust layers on specimens at velocities of 1, 2, and 5 meters per minute.

## PRESENTATION OF THE DATA

The essential information from the various experiments is shown in Table III by the average loss $\bar{X}$, the average standard deviation $\bar{\sigma}$, and the number $n$ of observations.[2] The estimated standard deviation $\sigma'$ of the universe is also given in the table, since it is a necessary step in the calculation of the error of each average. The errors of the averages were used in the construction of the average loss-time curves.

### *An Interpretation of the Data:*

It is evident from Table III that as the loss of weight increases the standard deviation increases. There are exceptions, but the authors are of opinion that these exceptions arise from sampling errors. The averages representing the longest periods of exposure where the weight loss is greatest are probably in error by more than those representing shorter periods of exposure, assuming that the number of observations is the same. If one would approach more and more nearly the objective average loss for various time intervals, the number of observations taken for the longer periods of time should be considerably larger than the number taken for the shorter times.

The experimental averages from Table III are shown in Figs. 4 and 5. The curves representing the relationship between time and average loss of weight have been drawn so that they do not miss the observed averages by more than their errors (see Table III). These curves have been plotted on log-log and semi-log coordinates so that the nature of relationship can be discerned. The first (Fig. 4) shows that the loss is proportional to time for an initial period[1] and the second (Fig. 5) shows that the loss is proportional to the logarithm of time after the initial period. There may also be a transition period.

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[1] See E. S. Hedges, "Protective Films on Metals," D. Van Nostrand Co., New York City (1932), and numerous enlightening articles by U. R. Evans, "The Distribution of Corrosion," *Transactions*, Am. Electrochemical Soc., Vol. LXI, p. 441 (1932).

[2] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I (1933); also published in pamphlet form.

An examination of the curves on log-log coordinates (Fig. 4) shows that the duration of the period over which iron loses weight in proportion to time decreases as the velocity is raised. It is not easy to estimate the duration of this period exactly, but the result of an effort to do so is shown in Fig. 6. The relationship between *duration of the initial period and velocity* is apparently hyperbolic. The curve shows that the loss is proportional to time for relatively long periods at low velocities and proportional to time only for very short periods at high velocities.

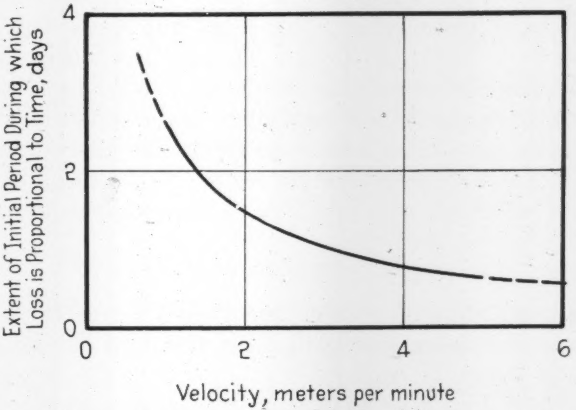

FIG. 6.—Relation Between Duration of Initial Period and Velocity.

It will be seen that the slope of the portion of the loss-time curves which is a straight line on semi-log coordinates (Fig. 5) is not the same at the three velocities. The lowest velocity has given the steepest curve. If the curves are extended, they will cross. Thus, iron starts to lose weight more rapidly at high velocities than it does at low velocities, but in the end it may lose less weight at the high velocities than at the low. There are, of course, hydraulic difficulties to be overcome if precise values of loss of weight are to be found for high velocities. It is also difficult to maintain a continuous flow of water so that losses may be determined accurately for long periods of exposure. These are the practical obstacles which must be overcome if the curves shown in this paper are to be extended.

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[1] Any straight line which passes through the origin will be a straight line on log-log coordinates with a slope of 1 (a 45-deg. line). In fact, any exponential which passes through the origin will be a straight line on log-log coordinates and the slope of the curve on log-log coordinates is the exponent. Thus, the curve $y = x^{\frac{1}{2}}$ is a straight line on log-log coordinates with a slope of $\frac{1}{2}$ (nearly a 30-deg. line).

The information which has been published by the Research Laboratory of Applied Chemistry at the Massachusetts Institute of Technology[1] either shows the fact or indicates the distinct possibility that the rust formed at a very high velocity is completely protective. Similarly, many of the data which have been reported from immersion tests on iron in stagnant or slowly moving water indicate that the loss is proportional to time.[2] Qualitatively, at least, these observations are not in disagreement with the evidence herein.

The material which has been presented in this paper is the result of loss of weight measurements made on iron specimens exposed to "hard" water. The rust formed on these specimens contains an appreciable quantity of calcium carbonate, which may have such an influence that the results are peculiar to these conditions. It would be very gratifying to learn that these results are general ones, but the authors believe that generalization would be unsafe if based on the limited data which are now available from miscellaneous sources.

## SUMMARY

1. The scattering of observations becomes greater as the loss of weight increases; more specifically, the standard deviation increases as the loss of weight increases.

2. Initially, iron loses weight in proportion to time.

3. The duration of the initial period decreases as the velocity is raised and is probably a hyperbolic function of the velocity.

4. After the initial period, during which the loss of weight is proportional to time, the specimens lose weight in proportion to the *logarithm of time* under the conditions of these experiments.

5. Iron specimens exposed to water at high velocity lose more weight for a time than those exposed at low velocity. After a month or so, the logarithmic curves are quite likely to cross, so that in the long run the specimens exposed at the higher velocities will probably lose less weight than those exposed at the lower velocities.

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[1] H. O. Forrest, B. E. Roetheli and R. H. Brown, "The Initial Corrosion Rate of Steels," *Industrial and Engineering Chemistry*, Vol. 22, p. 1197 (1930).

"Products of Corrosion of Steel," *Industrial and Engineering Chemistry*, Vol. 23, p. 650 (1931).

[2] R. F. Passano, "The Nature of the Corrosion-Time Relationship of Iron," presented at U. S. Bureau of Standards Third Corrosion Conference, March 30–31, 1933.

6. Continued association with a plan of investigation that has been based on the principles of statistical control (variation within limits) has only deepened the authors' belief in the wisdom of such a plan.[1] It was not easy at the beginning to understand just what difference there might be in the value of data produced by controlled and uncontrolled experimental conditions. We have found out from experience and the assurance that one gains from a knowledge that the conditions were controlled is unmistakable.

*Acknowledgments:*

The authors wish to express their appreciation of the unselfish cooperation which has been given by Messrs. O. A. Fritzsche and J. C. Young, of these laboratories, over the period when these observations were made. The analyses shown in the text were made by members of our chemical laboratory staff and we are grateful to them for the assistance which they have given.

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[1] W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., New York City (1931).

# DISCUSSION

MR. U. R. EVANS[1] (*presented in written form*).—The curves published by the authors are of the greatest value in showing the time-corrosion relations obtaining under conditions where "*the specimens corrode all over.*" The authors are extremely wise to italicize the words indicated. One must not forget that the most dangerous type of corrosion is that in which the attack is not general but localized on a few small points, the larger part remaining unattacked. In such case, the time-corrosion relations may well be different. This warning is not intended as a criticism; but it is rather intended to forestall criticism of a destructive nature.

The authors raise the question as to whether the results are valid, not only for the particular hard water used, but for other waters as well. There is no doubt that the rusting reactions in a water of this kind, containing numerous calcium, magnesium and sodium salts, are most complicated.[2] Even the action upon iron of a simple calcium bicarbonate solution under stagnant conditions is complicated, and the reactions go through several stages. Whether the stages are the same in running water is not quite certain, although the presence of calcium carbonate as an essential product of the rust certainly suggests that probably this is broadly true. But whatever view is held on details, the broad principle emerging from the work of recent years on corrosion, is that if the final corrosion product formed directly or indirectly from (*a*) anodically formed iron salt, (*b*) cathodically formed alkali, and (*c*) oxygen, is precipitated *at a sensible distance* from the metallic surface, *as a loose non-protective rust,* there will be *no retardation* of attack; whereas if this triple-interaction occurs *within the range of physical attractive forces,* so that a *clinging film* is produced, there may be protection and the velocity of attack *will fall off with the time.* Now, *ceteris paribus,* the "probability" that the "triple interaction" (whether it be direct or proceeding by stages) shall take place within range of physical attractive forces will increase with increase of oxygen supply, and with increasing uniformity of composition of the water washing the surface; that is, the probability will increase with the water velocity, provided that the "type" of flow is not thereby affected. The

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[1] Cambridge University, England.

[2] U. R. Evans, *Journal,* Soc. Chemical Industry, Vol. 47, p. 57T (1928).

"expected" value of the time needed for a fraction $\alpha$ of the surface at all points to become covered with a protective film will therefore decrease with the water velocity, $\alpha$ being defined as the fractional covering needed to cause "appreciable" departure of the time-corrosion curve from the straight course.[1] It is, therefore, quite to be expected that the "sensibly linear" portion of the corrosion-time curves shall continue for a long time at low water velocities and for a very short time at high water velocities. This argument makes no assumption as to the exact nature of the electrochemical process and the resulting precipitation and replacement, and therefore it seems not unreasonable to hope that the conclusions drawn may be at least fairly general.

MESSRS. E. C. GROESBECK[2] AND L. J. WALDRON[3] (*presented in written form*).—There is now in progress, at the U. S. Bureau of Standards, a study of the relationship existing between the velocity of flow and loss in weight when ferrous materials are completely submerged in flowing water of controlled pH value, oxygen concentration and temperature. An apparatus of the gravity flow type is being used for this study, in which the water is made to pass through a series of vertically arranged glass tubes at predetermined flow rates and past sheet specimens of the metal under test suspended in the water stream. The development of this testing apparatus was made without the knowledge that any similar type of apparatus was in use or contemplated elsewhere. Apparently, this general similarity in testing apparatus indicates a realization that more reliable results may be secured with this type of apparatus than with pre-existing types. The start of this work at a recent date does not permit the formation of any definite conclusions at this time.

The authors admit that their data are based on the use of a "hard" water in a particular locality, and that the resulting rust products may have exerted an influence on them. A confirmation of the general laws set forth in some of their conclusions by test results obtained with other types of water should form a valuable contribution to a better understanding of this subject. The control in temperature of the tap water derived from certain sources of city water supply would seem to require special consideration.

The results obtained in some unpublished work at the Bureau of Standards confirm the authors' findings that an increase in loss of weight produces an increase in the extent of the scatter in the plotted data. This appears to indicate the importance of the effect which the velocity of flow has on the liquid film surrounding the exposed metal surface. An increase in velocity should produce a thinning of the liquid film and thus promote the diffusion of oxygen to the metal surface per unit of time. Furthermore, a thin liquid film is influenced to a greater extent by the pH value, oxygen concentration and temperature of the surrounding solution, than a relatively thick one. Turbulence is set up in the water stream if the velocity exceeds a certain value. Innumerable oxygen concentration cells may be formed on the metal surface by this turbulence, and hence the difficulty of obtaining check results would be increased. The velocities reported by the authors, namely 5 meters and less, should produce a water flow that would be within the zone of straight-line flow.

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[1] Obviously no curve is absolutely straight over a finite length. In practice it is customary to neglect values of $d^2y/dx^2$ below a certain value, only regarding the departure from linearity as "sensible" when this value is exceeded.

[2] Metallurgist, U. S. Bureau of Standards, Washington, D. C.

[3] Assistant Metallurgist, U. S. Bureau of Standards, Washington, D. C.

In the Bureau's work, very satisfactory results have been secured by using the electrolytic method for cleaning the specimens. In this method, the specimen is made the cathode in an electrolyte, which is a 10 per cent sulfuric acid solution in this case, and a current density of about 10 ma. per sq. cm. is employed. The cleaning losses obtained by this method have been lower than those by the acid-cleaning method. The successful use in England of the electrolytic cleaning method with certain sodium salts used as electrolytes, has been reported.[1]

MR. J. R. BAYLIS.[2]—I should like to ask the author the length of time between the cleaning of the specimens. Did I understand that the specimens were cleaned occasionally during the test?

MR. R. F. PASSANO.[3]—The data in this paper are data representing continuous exposure to the conditions described. There was no interruption in the experiments while they were in progress. The specimens were removed from test at the end of the stated intervals (15 days, for example), and, as soon thereafter as possible, were immersed in hydrochloric acid containing an inhibitor to clean off the accumulated rust so the loss of weight for the interval could be determined. Each experiment for each interval was started from "scratch" with freshly prepared specimens.

MR. BAYLIS.—I misinterpreted that part of the paper, and formed the impression that the specimens were cleaned at certain intervals as the test progressed.

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[1] First Report of the Corrosion Committee to the Iron and Steel Industrial Research Council, British Iron and Steel Inst., p. 207 (1931).

[2] Physical Chemist, Department of Public Works, Chicago, Ill.

[3] Research Laboratories, The American Rolling Mill Co., Middletown, Ohio.

The water in which these tests were made is an extremely hard water, and it is believed that film formation had more to do with slowing up the rate of corrosion than anything else. The authors state that the water contained 334 parts per million of calcium carbonate and that the pH value was 7.5. If the water contained no magnesium, such a high-calcium carbonate concentration at this pH value would mean that the water was highly supersaturated with this compound. Magnesium occurs in nearly all natural waters, and it is likely that the amount occurring in the water used for conducting the tests was enough to cause the water to be just at saturation equilibrium with the calcium carbonate.

Corroding iron is a very good water softener. The iron combines with both the free $CO_2$ and the $CO_2$ in the bicarbonate alkalinity, and changes the calcium bicarbonate to calcium carbonate. This produces a supersaturation of calcium carbonate and causes it to precipitate. Also the ferrous carbonate will precipitate, so there will be two compounds tending to form a film on the iron surface in addition to the iron oxide precipitate. It is believed that the corrosion rate would have been nearer uniform if a very soft water had been used, though films formed by the iron precipitate likely would have retarded the corrosion quite materially. When very hard water is brought in contact with a large iron surface such as lathe turnings or iron filings the calcium carbonate will be precipitated within a very short time. The calcium occurring as calcium sulfate or calcium chloride will not be precipitated with corroding iron. It is believed that the falling off in corrosion rate can be attributed to films formed by calcium carbonate and ferrous carbonate.

MR. I. C. MATTHEWS.[1]—There is another factor that might be taken into consideration with the present discussion. That is, the ferric or ferrous sulfate present will precipitate organic matter which may also add to your film formation. A paper[2] presented before the American Electro-Platers' Society brought this out.

MR. PASSANO.—When I came to the meeting, I did not have sufficient assurance in the facts which our analyses of rust indicated to justify in my own mind a discussion of their composition. The things which Mr. Baylis has said are in such agreement with the indications of our analyses that a portion of my original reticence has been overcome.

At present we are stripping rust for analysis from specimens which have been in test at 0.5 meter per minute in very cold hydrochloric acid. The action of the acid on the iron causes some of the iron to go into solution as ferrous chloride and the hydrogen evolved in this process is available for the reduction of some of the ferric chloride already in solution. Without additional knowledge of the correction which must be made on this account, I do not believe that I should attempt to give quantitative results.

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[1] Research Chemical Engineer, Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

[2] F. J. Liscomb, "Impurities in Nickel Solutions," presented before meeting of American Electro-Platers' Society, June 27–30, 1933.

Inasmuch as the rust on the specimens has much water entangled in it and is put into solution in hydrochloric acid in this condition, we do not know the original weight of it and so at this point can express our results only in terms of weight. The amount of CaO in the rust increases with time; the amount of FeO increases with time and is about two or three times the CaO; the amount of $Fe_2O_3$ increases with time and is about four or five times the FeO. The behavior of the rust in the acid indicates the presence of $CO_2$ in appreciable quantity.

We have also taken some samples of rust mechanically and these we have dried in deoxygenated nitrogen. We find $SiO_2$, $CO_2$ from a carbonate and $H_2O$ (probably in the rust as entangled water, water of hydration and combination).

Using the data from these two separate analyses, we find that there is nearly enough $CO_2$ present to satisfy both the CaO and the FeO. If we consider the $SiO_2$ as being in combination with the CaO, the rust is a mixture of calcium silicate, calcium carbonate, ferrous carbonate[1] and ferric hydroxide. It is interesting that on these assumptions the calculated total is nearly 100 per cent of the weight of rust obtained by drying the mechanically taken sample at room temperature in deoxygenated nitrogen. This is surprising because the total weight of the dried rust sample is often less than 0.5 g.

From this you will gather that data from actual analyses are in agreement with Mr. Baylis' postulation from his previous knowledge on the existence of calcium and ferrous carbonates in the rust.

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[1] There may also be ferrous hydroxide present.

# CORROSION RESISTANCE OF STRUCTURAL ALUMINUM

By E. H. Dix, Jr.[1]

## Synopsis

The mechanical testing of thin sheet specimens, as a means of evaluating the relative corrosion resistance of metals, is being used by an increasing number of investigators. It is one of the methods being employed in the atmospheric testing program of Subcommittee VI, of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. Such data, however, do not form a satisfactory basis for formulating a true conception of the mechanical permanence of structural shapes which, of course, are much thicker in section. To bridge this gap, in the case of aluminum alloys, the Aluminum Research Laboratories have undertaken an extensive series of tests involving outdoor and accelerated corrosion exposures of full-size structural shapes in comparison with sheet and plate specimens.

These tests have shown that the corrosion of the duralumin type alloy, 17ST, is "self-stopping" and hence, tension tests made on thin specimens give an entirely misleading conception of the structural stability of thicker sections. Beam and column tests performed on full-size sections show no decrease in the load-supporting capacity of the 17ST shapes after exposure to severely corrosive conditions which caused marked losses in the mechanical properties of thin-sheet specimens of the same alloy.

## Introduction

The strong aluminum alloys, of the duralumin type, owing to their high strength per unit weight and good corrosion resistance have found extensive use for structural parts of airships and airplanes. In the early days of aircraft construction, little was known about the corrosion resistance of duralumin, and in consequence corrosion difficulties were sometimes encountered, in the thin sections used, through lack of even minor precautionary measures. Through the experience gained during the past twenty years or more, the uniformity of the corrosion resistance of the product has been greatly improved by advancements in fabricating technique, and adequate protective measures have been evolved for severe conditions of service.

Aluminum is now finding rapidly increased application in steam and electric railways, marine, bus and truck construction. It is but a step from these applications to such stationary structures as bridge floors, long-span bridges and structural parts of the upper floors of tall buildings, where high strength per unit weight is economical. As an example of the large sections which are now produced for such applications may be mentioned 10-in. ship channels 85 ft. long. Such shapes are heat treated in electric furnaces of a high degree of temperature uniformity and are rapidly quenched in high-pressure water sprays.

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[1] Chief Metallurgist, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

## MATERIALS

In the tests to be discussed, a duralumin-type alloy of the Aluminum Company of America, containing nominally 4 per cent copper, 0.5 per cent manganese, and 0.5 per cent magnesium, has been selected as typical of a structural aluminum alloy (see Table I). This alloy in the annealed temper has a high degree of workability and after a solution heat treatment followed by a rapid quench, attains a strength approximately equivalent to that of structural steel. The symbol 17ST is used to designate this alloy in the heat-treated temper. Immediately after quenching, it is still relatively soft so that it is often desirable to do the more severe forming operations within an hour or two after quenching. The alloy ages naturally at room temperature and attains substantially its maximum properties in about four days, after which there is practically no further change. Other aluminum alloys are available having yield strengths 30 to 50 per cent higher than alloy 17ST. For applications which do not require as high a strength as alloy 17ST, a saving in cost may be effected by employing a non-heat-treatable aluminum alloy.

TABLE I.—DESCRIPTION OF MATERIALS TESTED.

| Item[a] | Shape | Nominal Size | Area, sq. in. | Weight, lb. per ft. | Chemical Analysis, per cent | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Silicon | Iron | Copper | Manganese | Magnesium | Aluminum |
| | | | | ALUMINUM ALLOY (17S) | | | | | | |
| 1 | I-Beam[b] | 5 in. by 0.210 in. | 3.00 | 3.63 | 0.40 | 0.44 | 3.90 | 0.64 | 0.58 | Bal. |
| 2 | Angle | 6 in. by 4 in. by $\frac{3}{8}$ in | 3.72 | 4.52 | 0.15 | 0.50 | 4.31 | 0.65 | 0.47 | Bal. |
| 3 | Sheet | 0.064 in | .... | .... | 0.36 | 0.37 | 3.76 | 0.65 | 0.43 | Bal. |
| 4 | Channel | 6 in. by 0.2 in | 2.40 | 2.91 | 0.21 | 0.55 | 4.30 | 0.69 | 0.51 | Bal. |
| 5 | Sheet[c] | 0.064 in | .... | .... | 0.53 | 0.49 | 4.04 | 0.58 | 0.52 | Bal. |
| | | | | | 0.55 | 0.57 | 3.86 | 0.59 | 0.54 | Bal. |
| | | | | | 0.54 | 0.51 | 4.08 | 0.57 | 0.55 | Bal. |

[a] Items 1, 2 and 3 used in alternate immersion test.
Items 4 and 5 used in outdoor exposures.
[b] Extruded; all others rolled.
[c] Item 5 represents three different mill lots of sheet.

## TEST METHODS

Relatively thin sections are used for aircraft structures, and hence most of the early corrosion tests of duralumin have been conducted on relatively thin sheet. This procedure has been continued largely because easily measurable effects of corrosion are obtained in a relatively short time. Now, however, in view of the newer applications employing thicker sections, it is highly important to have a correlation between the results obtained on thin sheet and on thicker sections.

The structural engineer is interested primarily in the possible effect of corrosion in reducing the strength of structures and hence from his viewpoint, the most satisfactory method of evaluating corrosion resistance is to compare the strength of structural parts before and after various corrosive exposures.

TABLE II.—RESULTS OF TESTS ON BEAMS AND COLUMNS SUBJECTED TO ALTERNATE IMMERSION IN A $3\frac{1}{2}$ PER CENT SEA-SALT SOLUTION.

| | Original Properties | After Exposure for 1 yr.[a] | | After Exposure for $2\frac{1}{2}$ yr. | |
|---|---|---|---|---|---|
| | | Actual | Per Cent Change | Actual | Per Cent Change |
| 5 IN. 17ST I-BEAM<br>Tension Specimens 0.214 in. Thick from Web[b] | | | | | |
| Tensile strength, lb. per sq. in. | 58 700 | 56 750 | −3 | 55 850 | −5 |
| Yield strength, lb. per sq. in. | 41 350 | 40 250 | −3 | 40 400 | −3 |
| Elongation in 2 in., per cent | 19.3 | 15.0 | −22 | 14.8 | −23 |
| Simple Beam Loaded at Third Points, $67\frac{7}{8}$-in. Span | | | | | |
| Maximum beam load, lb | 20 670 | 19 700 | −5 | 20 950 | +1 |
| 6 BY 4 BY $\frac{3}{8}$-IN. 17ST ANGLE<br>Tension Specimens 0.375 in. Thick from Leg[b] | | | | | |
| Tensile strength, lb. per sq. in. | 53 150 | 52 180 | −2 | 52 380 | −2 |
| Yield strength, lb. per sq. in. | 30 600 | 30 500 | 0 | 30 850 | +1 |
| Elongation in 2 in., per cent | 22.5 | 16.0 | −29 | 14.3 | −36 |
| Flat End Columns 72 in. Long | | | | | |
| Maximum column load, lb | 72 000 | 77 600 | +8 | 75 500 | +5 |

[a] 2274 cycles.

[b] Tensile properties are average of two tests on standard A.S.T.M. plate specimens comprising full thickness of section. Corroded specimens cut from shapes after exposure.

[c] 7762 cycles.

The use of tension tests of standard sheet specimens to evaluate the corrosion resistance of metals and alloys was first described by E. Blough in 1925,[1] and has been used by other investigators, notably

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[1] E. Blough, "The Evaluation of Corrosion Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 162 (1925).

H. S. Rawdon[1] to study the corrosion resistance of duralumin sheet. It is also one of the methods being employed in the atmospheric corrosion program of Subcommittee VI, of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

However, to predict accurately the life of a structure, tests should be made on full-size sections after selected corrosion exposures, rather than attempting to extrapolate the results obtained on thin tension specimens. It is the purpose of this paper to describe what are believed to be the first corrosion tests performed on full-size structural shapes, and to compare the more reliable results thus obtained with tests on tension specimens individually exposed to similar corrosive conditions.

TABLE III.—RESULTS OF OUTDOOR EXPOSURE TESTS.

|  | Original Properties | Per Cent Change After Exposure |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|
|  |  | New Kensington, Pa. |  |  | Point Judith, R. I. |  | Edgewater, N. J. |  |
|  |  | 1 yr. | 2 yr. | 5 yr. | 1 yr. | 2 yr. | 1 yr. | 2 yr. |
| TENSION TESTS ON SPECIMENS FROM 6-IN. CHANNELS[a]<br>17ST, 0.220 in. Thick |  |  |  |  |  |  |  |  |
| Tensile strength, lb. per sq. in. | 58 100 | −2 | .... | .... | −1 | .... | −2 | .... |
| Yield strength, lb. per sq. in. | 35 200 | −1 | .... | .... | −1 | .... | −1 | .... |
| Elongation in 2 in., per cent | 21.2 | −6 | .... | .... | −6 | .... | −3 | .... |
| TENSION TESTS ON SHEET SPECIMENS[b]<br>17ST, 0.064 in. Thick |  |  |  |  |  |  |  |  |
| Tensile strength, lb. per sq. in. | 62 560 | −5 | −7 | −7 | −13 | −14 | −5 | −5 |
| Yield strength, lb. per sq. in. | 35 330 | −4 | −7 | −7 | 0 | −5 | −6 | −1 |
| Elongation in 2 in., per cent | 21.1 | −15 | −13 | −15 | −55 | −55 | −30 | −21 |

[a] Tensile properties obtained on standard A.S.T.M. plate specimens comprising full thickness of web. Original properties are average of three tests. Losses from corrosion were determined by testing six specimens cut from channels after exposure. Actual exposure periods: New Kensington, 1.32 yr.: Point Judith, 0.77 yr.; and Edgewater, 1.23 yr.

[b] Tensile properties obtained on five standard A.S.T.M. sheet specimens of each of three lots of 17ST. Specimens were machined before exposure.

Salt-water or a salt-laden atmosphere are recognized as among the most corrosive conditions encountered in outdoor usage of aluminum alloys. For this reason, a $3\frac{1}{2}$ per cent sea-salt solution was selected as the corrosive medium to be used in the accelerated tests. Tanks containing the structural shapes were filled with the solution approximately every two hours, and then emptied. The time necessary for the complete filling and draining of the tanks was seven to ten minutes.

Table II presents the data obtained in the alternate immersion test. The tensile properties were obtained from standard A.S.T.M.

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[1] H. S. Rawdon, "The Correlation of Laboratory Corrosion Tests with Service: Weather-Exposure Tests of Sheet Duralumin," *Transactions*, Inst. Metals Division, Am. Inst. Mining and Metallurgical Engrs., p. 220 (1929).

plate tension specimens, including the full thickness of the sections, cut from the shapes after the several corrosion periods. The I-beams were tested as simple beams loaded at the third points with a span of approximately 68 in. The angles were tested as flat-end columns approximately 72 in. long. The data obtained from outdoor exposures are given in Table III.

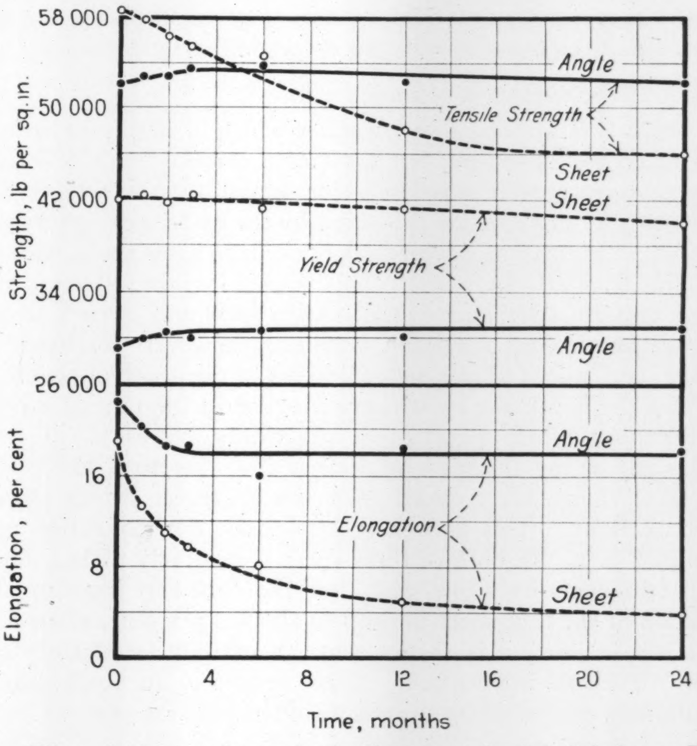

FIG. 1.—Effect of Alternate Immersion in 3½ per cent Sea-Salt Solution on Tensile Properties of Standard A.S.T.M. Specimens Comprising Full Thickness of Section.

Specimens from angles were exposed as blanks 1 in. wide by 0.375 in. thick, edges machined after corrosion. Specimens of sheet, 0.064 in. thick, were machined before exposure.

## DISCUSSION OF DATA

*Beam and Column Tests:*

The full-size beam and column tests indicate that there has been no significant loss in load-supporting capacity, even after 2½ yr. exposure to this severely accelerated corrosive condition. Small losses in strength and elongation were indicated by specimens cut from these shapes after exposure but the losses were no greater after $2\frac{1}{2}$ yr. than after 1 yr.

*Tension Tests on Individually Exposed Specimens:*

The results obtained on individually exposed specimens for pilot tests are shown graphically in Fig. 1. The comparison between the effect of corrosion on the sheet, 0.064 in. thick, and the specimens cut from the angle, 0.375 in. thick, shows very conclusively the great importance of thickness in resisting deterioration by corrosion.

*Corrosion Characteristics of Aluminum Alloys:*

Corrosion is a surface phenomenon which is largely independent of the thickness of section, but a pit of a certain depth will have a relatively much greater effect in a thin section than in a thicker one. Aluminum and its alloys are protected by the oxide coating formed by corrosion. Penetration, however, does occur in some spots, where the oxide film is relatively weak. This causes localized stresses which prevent uniform stretching in the tension tests and cause a decrease in elongation. Such corrosive action is, however, relatively self-stopping, and indeed actual measurements of the depth of penetration show that the depth is a logarithmic function of the time of exposure. The depth at which penetration practically ceases is, of course, independent of the thickness of section and, therefore, thin sections have their elongation reduced to a much greater extent than the thicker sections. This is well illustrated in the elongation curves shown in Fig. 1. In both of these there is relatively little additional loss in two years over one year, but the corrosion has caused the elongation of the thin sheet to drop to about 4 per cent, whereas that of the heavier section is 18 per cent, an entirely satisfactory figure for any structural application. These results of an accelerated test are strikingly confirmed by the results of the outdoor exposure tests.

*Outdoor Exposure Tests of Sheet Specimens:*

It is evident that since no measurable losses were obtained in beam and column tests on I-beams and angles of alloy 17ST after $2\frac{1}{2}$ yr. exposure to an extremely accelerated corrosive condition, it will require very extended periods of outdoor exposure before measurable changes can be obtained from the effects of such mildly corrosive conditions. Therefore, for the present, data on the behavior of alloy 17ST in outdoor exposures must be limited to tests of thin-sheet specimens. Full-size structural shapes are undergoing exposure and will furnish interesting data at a later date. The results of tests on thin specimens which have been exposed for 1 and 2 yr. and, in one case, 5 yr., at three different outdoor locations are given in Table III. New Kensington, Pa., and Edgewater, N. J., represent a typical inland industrial atmosphere and an industrial atmosphere combined with weak seacoast influences, respectively. The frames supporting the specimens were inclined at 45 deg. to the horizontal and faced south. The specimens were mounted so that their axes were in the plane of the frame and their flat surfaces perpendicular to this plane. At these two stations the frames were mounted on the roofs of plant buildings. The site at Point Judith, R. I., representing severe seacoast exposure, is located about one hundred yards from the surf, on a point with the ocean on two sides and a salt marsh on the third. The frames were raised about three feet from the ground at this station.

These outdoor exposure tests confirm in a very striking manner several indications obtained from the more accelerated exposures. Most important is the fact that at all three locations the thin-sheet specimens showed no greater losses in properties after 2 yr. than after 1 yr., and at New Kensington there has been no increase in the losses after 5 yr. The results of longer exposures at all stations will be available at later dates. This confirms the deduction that the corrosion of aluminum alloys is self-stopping under a variety of outdoor exposure conditions as well as in alternate immersion. A comparison between the results obtained on the thin-sheet specimens and the thicker plate specimens cut from channels again emphasizes the importance of thickness in resisting impairment of mechanical properties. It is also of interest to observe that in general the yield strength of the alloy is affected to a lesser extent than the tensile strength. This is particularly noticeable in the specimens exposed to the extremely severe corrosive conditions at Point Judith.

## CONCLUSIONS

The results presented in this paper lead to the following conclusions regarding the corrosion resistance of structural aluminum:

1. The corrosion of the duralumin type alloy, 17ST, is practically self-stopping and the depth of penetration is independent of the thickness of the section. Therefore, tension tests on thin specimens after exposure to short-time test conditions give an entirely misleading conception of the corrosion resistance of heavier sections, unless properly interpreted. Such tests, however, are of considerable value when correlated with service experience.

2. In studying the changes in mechanical properties of tensile specimens after exposure to various corrosive conditions, it is observed

that the yield strength is less affected than the tensile strength and that the losses in tensile and yield strengths and elongation tend to approach a limit below which they do not drop appreciably, even when the exposure period is doubled, and that this limit is higher the thicker the section.

3. Sections 0.2 in. and thicker, show losses so small as to be unimportant structurally even after extended exposure to very severely corrosive conditions.

4. No appreciable losses in load supporting ability in beams and columns occurred under severely corrosive conditions which caused marked losses in mechanical properties of thin-sheet specimens of the same alloy.

*Acknowledgment:*

The author desires to acknowledge with thanks his indebtedness to Dr. W. L. Fink, Dr. L. J. Weber and Mr. J. A. Nock, Jr., under whose direction the corrosion exposures were carried out; to Mr. R. L. Templin and his associates, Messrs. E. C. Hartmann and M. Holt, who directed the mechanical testing; and to Mr. H. V. Churchill and his staff for the chemical analyses.

# DISCUSSION

MR. L. B. TUCKERMAN[1] (*presented in written form*).—Mr. Dix's paper forms a welcome addition to our knowledge of the corrosion resistance of alloys of the duralumin type, since it confirms impressions which have been gained from studies of thinner materials.

The tests at the U. S. Bureau of Standards, both the extensive, systematic corrosion tests carried out by the Division of Metallurgy[2] (on material 0.063 in. thick) and the periodic inspection tests on aircraft in service carried out by the Engineering Mechanics Section have been limited to sheet and tube with thickness ranging from 0.008 in. to 0.080 in. They have indicated that, from a structural standpoint, the corrosion becomes less serious as the thickness is increased. This indication, however, has been far from conclusive since the materials of different thicknesses were, in general, from different sources and not uniform either in chemical composition or in metallurgical treatment. Mr. Dix's work not only confirms this indication but places it upon a much surer footing by comparing materials whose chemical composition and metallurgical treatment are fully known. The Bureau's tests have also indicated that the corrosion slows down with time, so that material which, under the most severe types of weather exposure, is still structurally sound after 18 months has shown no appreciable further deterioration in the following 3½ years.

I am wondering, however, whether we are quite ready to draw the conclusion: "1. The corrosion of the duralumin type alloy, 17ST, is practically self-stopping. . . ." I should not today hesitate to advocate the use of the heavier sections of duralumin type alloys under exposure conditions where, five years ago, I would have wondered whether their use was advisable. On the other hand, 3½ years with no appreciable further progress of corrosion seems hardly sufficient to ensure that 15 or 20 years might not show a measurable effect.

Although it is apart from the main subject of his paper, Mr. Dix might have pointed out the great progress made in the last ten years in producing duralumin type alloys which, even in thin sheet, are remarkably resistant to corrosion. The Alclad hull of the Navy Airship MC-2, only 0.01 in. thick, after four years of service has shown no measurable decrease in its structural properties, although exposed to the salt-laden air at Lakehurst, and plain duralumin with suitable heat treatment, 0.05 to 0.08 in. thick, shows similar good results. This leads us to expect that this type of light alloy will find ever broader fields of usefulness.

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[1] Assistant Chief, Division of Mechanics and Sound, U. S. Bureau of Standards, Washington, D. C.

[2] H. S. Rawdon, "Corrosion Embrittlement of Duralumin," *Technical Notes Nos. 282, 283, 284, 285, 304, and 305*, Nat. Advisory Committee Aeronautics (1928 and 1929).

MESSRS. H. S. RAWDON[1] AND W. H. MUTCHLER[2] (*presented in written form*).—The results presented by Mr. Dix should be of very great interest to all actual and prospective users of aluminum alloys as structural materials for purposes in which atmospheric corrosion must be considered as an important factor determining the useful service life of the material. The results tie in very nicely with data which have been secured by the Bureau of Standards in a rather extensive series of exposure tests of aluminum alloys covering the past five years. This investigation covered a variety of aluminum alloys, all in sheet form (No. 14 gage), the effect of various heat treatments on the corrosion resistance of the materials, and the general usefulness and reliability as a protective measure of a great many surface coatings and related surface treatments. Reference will be made here only to one material, the well-known 17S alloy, in two conditions. The results are summarized in the accompanying Fig. 1. One set of curves shows the properties of the material in the heat-treated condition (aged at room temperature after being

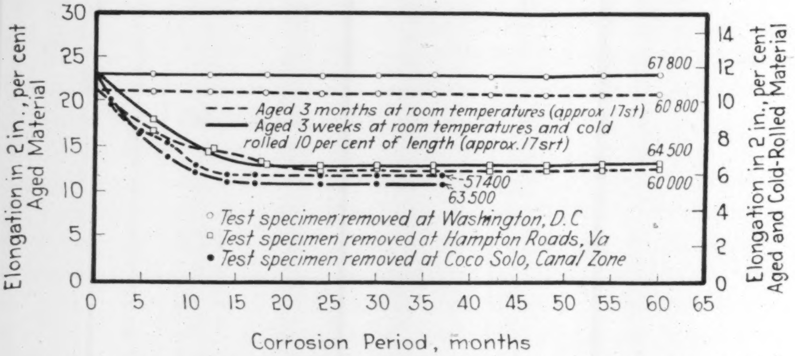

FIG. 1.—Effect of Weather Exposure upon 14-gage Sheet Duralumin (17S).

All specimens given a solution heat treatment 15 minutes at 505 C.

NOTE.—Figures at ends of curves are average tensile strength, pound per square inch, values of all specimens on the horizontal section of the curve.

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[1] Chief, Division of Metallurgy, U. S. Bureau of Standards, Washington, D. C.

[2] Junior Metallurgist, U. S. Bureau of Standards, Washington, D. C.

quenched in cold water from 505 C.), and the second shows the properties of similar material which was strengthened by being cold rolled somewhat after being aged. The exposure tests were carried out under widely differing climatic conditions as illustrated by the three localities shown, Washington, D. C., Hampton Roads, Va., and Coco Solo, Canal Zone. The progressive changes in the properties of the materials as determined on tension specimens exposed continuously to the weather until the time of testing are summarized in the accompanying Fig. 1.

The marked change in properties occurred in the materials exposed in the marine atmosphere, the specimens exposed at Washington being practically unaffected during the 5-year period. A marked lowering in ductility occurred during the first 18 months after which a stable state was apparently reached. It is noteworthy that the tensile strength of the materials was not lowered in the same degree as was the elongation. For this particular alloy used under the conditions indicated, one should have no serious misgivings concerning the reliability in service. Alloys of other compositions and in different conditions of treatment should, of course, not be included in the particular case discussed.

MR. F. N. SPELLER[1] (*presented in written form*).—Strength tests which we made recently on pitted steel pipe about 0.3 in. thick confirm the results obtained by Mr. Dix on duralumin.

Several lengths of old steel gas mains 10 to 14 in. in diameter were cleaned and depth of pitting measured. The ends were then closed and hydrostatic pressure applied until the pipe fractured. The average calculated fiber stress at the bursting pressure was found to be practically equal to the expected stress of uncorroded pipe, where the pitting was not more than 40 per cent of the thickness. The pits were widely scattered and not closer than 3 or 4 in. from one another. Other tests made on new pipe in which pit holes were drilled confirmed these results. Evidently, as Mr. Dix has found, reduction in strength is not always a reliable measure of corrosion except in very thin metal.

MR. E. H. DIX, JR.[2] (*author's closure by letter*).—The author appreciates very much the discussions by Messrs. Tuckerman, Rawdon and Mutchler, and Speller. The data quoted by all of these gentlemen is very pertinent and aids greatly in amplifying the results of the original paper.

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[1] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.

[2] Chief Metallurgist, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

# A COMPARISON OF CERTAIN WHITE-METAL BEARING ALLOYS, PARTICULARLY AT ELEVATED TEMPERATURES

By C. E. Swartz[1] and A. J. Phillips[1]

## Synopsis

A study has been made of the physical properties of certain tin-base and cadmium-base bearing alloys, especially at elevated temperatures (up to 570 F., 300 C.). Practical tests of three different types were also included in the investigation.

In all the tests the cadmium-base bearing alloy showed properties equal to or superior to the tin babbitt. Since the solidus of the cadmium-base alloy is higher than that of the tin-base babbitt alloy the former was satisfactory for bearing purposes at temperatures above those at which the latter failed. While the compressive strength of the cadmium-base alloy is higher than that of the tin babbitt alloy, yet its hardness was not sufficient to injure the softest steel shaft. Further, the alloy is not brittle as shown by the elongation and reduction of area in tension, these values being of the same order as those of the tin-base alloy.

These physical test data were confirmed by practical tests. For example, a six-cylinder test motor showed that the cadmium-base alloy continued to function after the tin-base babbitt alloy had failed due to temperature.

## Introduction

Some interest has been shown recently in white-metal bearing alloys especially for use in automotive motors. This increased interest is due, at least in part, to the present trend toward higher compression, higher speed, and more sustained speed of motors, necessitating the use of a bearing composition which will stand this more severe service.

As will be noted in the discussion of papers on white-metal bearing alloys, there is considerable difference of opinion as to which physical properties are related and as to the correlation of these properties to results obtained in actual commercial installations. The physical properties most commonly studied have been strength in compression and hardness.

Freeman and Woodward[2] and Freeman and Brandt[3] made extensive tests on the compressive strength of both lead- and tin-base bearing alloys. Ellis and Karelitz[1] have determined compressive strength and hardness at temperatures up to 285 F. (140 C.). Clamer,[2] in addition, studied the tensile properties of alloys used in railway car journals. Herschman and Basil[3] have reported hardness, compressive strength, data on wear resistance, and repeated and single impact, as well as service tests.

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[1] Central Research Laboratory, American Smelting and Refining Co., Maurer, N. J.

[2] J. R. Freeman, Jr., and R. W. Woodward, "Some Properties of White-Metal Bearing Alloys at Elevated Temperatures," U. S. Bureau of Standards *Technologic Paper No. 188* (1921).

[3] J. R. Freeman, Jr., and P. F. Brandt, "The Influence of the Ratio of Length to Diameter in the Compression Testing of Babbitt Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 150 (1923).

In the investigation here reported an endeavor was made, first, to select tests measuring physical properties recognized as of fundamental importance in predicting the adaptability of an alloy for use as a bearing under given conditions of load, temperature, etc.; second, to omit tests which are not commonly used or which would be covered equally well by service tests; and third, to outline definite service tests which would give a practical check on the results found in the tests selected.

Of the many properties a good bearing alloy should possess, the following are important: (1) An adequate strength in compression; (2) a softening point high enough to withstand any temperature likely to be encountered in practice; (3) a plasticity or ability to deform (creep) under load, to take care of alignment; (4) a ductility, or better, an absence of brittleness to withstand pounding and torque without cracking; (5) a sufficient but not excessive hardness; and (6) an "alloyability," that is ability to "wet," "tin," or bind itself chemically with any ordinary backing material.

Accordingly the following physical properties were among those investigated:

1. Compressive strength at room and elevated temperatures.
2. Tensile strength including elongation and reduction of area at room and elevated temperatures, these being considered as measures of ductility.
3. Creep strength at room temperature. So far as the authors are aware, no results on creep of bearing alloys have been reported. The ability of a shaft to align itself through deformation of the bearings supporting it would appear to depend as much on creep as upon any other property.
4. Hardness.
    - (*a*) Rockwell Hardness at room temperature.
    - (*b*) Brinell Hardness at room and elevated temperatures.
    - (*c*) Microhardness at room temperature.

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[1] O. W. Ellis, and G. B. Karelitz, "A Study of Tin-Base Bearing Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 49–50, MSP 50–11, p. 13 (1927–1928).

[2] G. H. Clamer, "Effect of Changes in Composition of Alloys Used by the American Railways for Car Journal Bearings," *Transactions*, Am. Inst. Metals, Vol. 9, p. 241 (1915).

[3] H. K. Herschman and J. L. Basil, "Mechanical Properties of White-Metal Bearing Alloys at Different Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 536 (1932).

5. Practical tests. In these tests, two alloys were run side by side or one alloy was replaced by another. Three different tests were made as follows:

(a) Low-speed light duty,
(b) High-speed heavy duty,
(c) Automotive duty.

Inasmuch as the wear tests of Herschman and Basil[1] agreed with their service tests, no wear tests were made in the present program. Since the various methods of impact testing do not appear to be standardized for bearing alloys, and since these values are rather closely related to hardness tests, these tests were also omitted from this investigation.

## MATERIALS

*Metals.*—All alloys used in these tests were made by the authors from metals of high purity; pure tin, refined antimony, electrolytic copper, electrolytic cadmium and pure sheet nickel.

*Alloying.*—Alloys were prepared by melting the constituents in a Dixon clay-graphite crucible with stirring, using a fused salt cover of zinc chloride to which small amounts of ammonium chloride were added from time to time. In the case of the cadmium-nickel alloys, cadmium was first melted, then nickel in thin sheets was introduced through the fused zinc chloride. Considering the great difference in the melting points of cadmium and nickel, alloying was surprisingly easy. In all cases the melting and alloying was done in a small electric resistance pot furnace. This allowed a careful control of the temperature so that overheating was easily avoided. In one case overheating of about 200 F. (110 C.) was purposely allowed. Neither the tin-base nor the cadmium-base alloy appeared to be injured.

*Casting.*—After alloying had taken place and the melt was stirred to insure homogeneity, the temperature was brought to 750 F. (400 C.) and the alloy cast into suitably shaped molds preheated to 212 F. (100 C.). Castings for tension specimens were $\frac{13}{16}$ in. in diameter and approximately $5\frac{1}{4}$ in. long. Compression specimens were taken from castings $1\frac{1}{4}$ in. in diameter and approximately $5\frac{1}{2}$ in. long. For rolled material such as was used in creep tests, castings were $\frac{3}{4}$ by $1\frac{1}{2}$ in. by approximately 11 in. long.

Micro-examinations indicated that the same casting conditions would produce satisfactory microstructure in both tin- and cadmium-base alloys.

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[1] Discussion by O. W. Ellis, of paper by H. K. Herschman and J. L. Basil, "Mechanica Properties of White-Metal Bearing Alloys at Different Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 556 (1932).

Of the various alloys studied only three will be discussed in detail.

1. Tin-base: 5.23 per cent copper, 6.2 per cent antimony, remainder tin.

2. Cadmium-base: (*a*) 3 per cent nickel, remainder cadmium; (*b*) 1.35 per cent nickel, remainder cadmium.

## TEST METHODS AND RESULTS

For the strength tests, a 60,000-lb. capacity Southwark-Emery Universal Hydraulic Testing Machine was used. Loads up to 3000 lb. were measured on a dial reading directly to 5 lb. Loads from 3000 lb. to 15,000 lb. were measured on a dial reading directly to 20 lb. The rate of specimen deformation was controlled by a careful manual adjustment of the throttle to maintain a head movement of 0.10 in. per minute as indicated by a dial gage reading directly to 0.001 in. In the compression tests, the dial was fastened to the anvil and actuated by the movement of the auxiliary head thus measuring almost exactly the absolute deformation of the specimen. In the tension tests the dial indicated the movement of the machine head and cannot be considered as measuring absolute specimen deformation. Since the tension tests were primarily used to measure ductility, absolute measurement of specimen deformation was considered unnecessary.

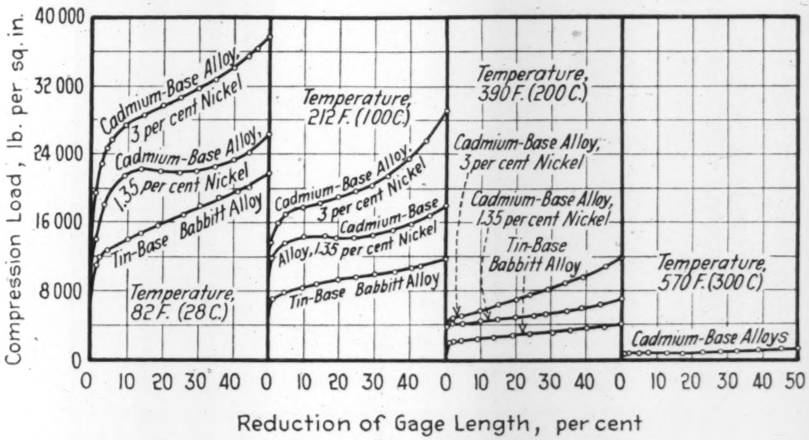

FIG. 1.—Compressive Stress-Strain Relationship.

*Compression Tests:*

Compression tests were made on the three alloys at 82 F. (28 C.); 212 F. (100 C.); 390 F. (200 C.); and 570 F. (300 C.). Elevated temperatures were maintained by surrounding the specimen, and anvil and auxiliary head of the testing apparatus with circulated oil held at the desired temperature by electric immersion heaters. The specimens were machined to 1 in. in diameter by 2 in. long. Figure 1 shows the results of the compression tests. All curves are plotted from the results of at least two test specimens. Only a few of the values obtained are shown on these curves.

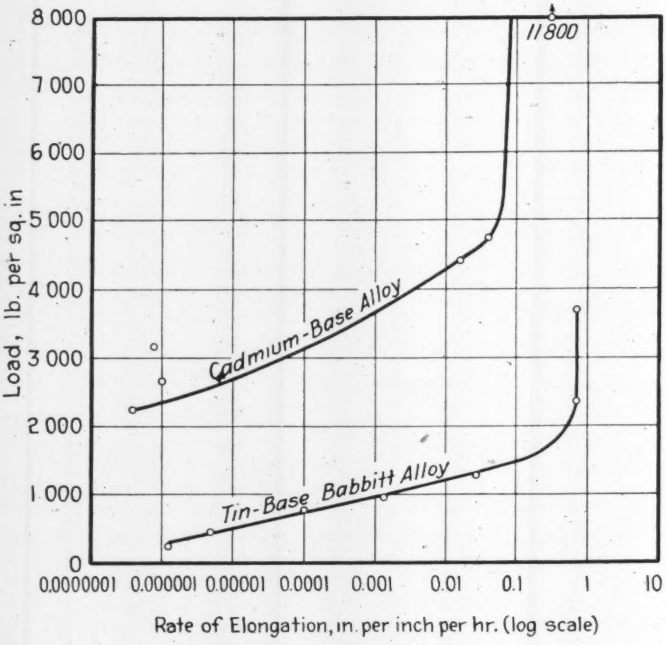

FIG. 2.—Creep Rate Curves.

*Tension Tests:*

Tension tests were made at the same temperatures as the compression tests. For the elevated temperatures, special extension grips were used in order to project into a furnace filled with oil. The desired temperature was maintained by immersion heaters and stirring. The furnace was sufficiently long to keep the specimen immersed in the oil during the entire test although extraordinary elongations took place in some cases. Standard threaded-grip specimens 0.505 in. in diameter with a 2 in. gage length were used. The yield point (determined only at room temperature) was defined arbitrarily as 0.5 per cent of the gage length or 0.01 in. in 2 in. Table I records the results of the tension tests on the three alloys.

*Creep Tests:*

In order to eliminate irregularities in the materials due to casting, only cold-rolled specimens were used. This is permissible in that it is only the relative creep rates which are of interest. Specimens were 1 in. wide, with a gage length of 8 in. A dead weight of approximately 45 lb. was used in all cases, the individual weights being known to within 0.01 lb. The load was varied by varying the thickness, between 0.01 and 0.05 in. on the cadmium-nickel specimens, and 0.02 to 0.17 on the tin-babbitt specimens. Figure 2 shows the creep curves of two of the alloys under discussion. Although the tests have not been carried far enough to determine actual values, the creep limit of the tin alloy appears to be in the neighborhood of 200 lb. per sq. in., while the cadmium bearing alloy has a creep limit of about 2000 lb. per sq. in.

TABLE I.—TENSION DATA ON BEARING ALLOYS.

| Composition | Temperature | Tensile Strength, lb. per sq. in. | Yield Point, lb. per sq. in. | Elongation, per cent | Reduction of Area, per cent |
|---|---|---|---|---|---|
| 5.23 per cent copper; 6.2 per cent antimony; remainder tin | 82 F. (28 C.) | 10 400 | 8 500 | 10 | 12 |
| | 212 F. (100 C.) | 5 900 | ...... | 24 | 38 |
| | 390 F. (200 C.) | 1 800 | ...... | 86 | 76 |
| | 570 F. (300 C.) | alloy molten at 475 F. (245 C.) | | | |
| 1.35 per cent nickel; remainder cadmium | 82 F. (28 C.) | 16 400 | 11 700 | 19 | 43 |
| | 212 F. (100 C.) | 10 500 | ...... | 36 | 52 |
| | 390 F. (200 C.) | 3 300 | ...... | 111 | 76 |
| | 570 F. (300 C.) | 600 | ...... | 162 | 78 |
| 3 per cent nickel; remainder cadmium | 82 F. (28 C.) | 22 900 | 16 800 | 6 | 6 |
| | 212 F. (100 C.) | 14 300 | ...... | 13 | 17 |
| | 390 F. (200 C.) | 3 300 | ...... | 65 | 42 |
| | 570 F. (300 C.) | 700 | ...... | 213 | 82 |

TABLE II.—BRINELL HARDNESS OF BEARING ALLOYS.

For the purpose of comparison, values on a typical high-lead alkaline-earth bearing alloy are shown. These results are taken from the manufacturer's published data.

| Composition | Brinell Numbers | | | | |
|---|---|---|---|---|---|
| | 82 F. (28 C.), 500-kg. Load | 212 F. (100 C.), 500-kg. Load | 300 F. (150 C.) | 390 F. (200 C.), 250-kg. Load | 570 F. (300 C.), 100-kg. Load |
| 5.23 per cent copper; 6.2 per cent antimony; remainder tin | 24.4 | 12.4 | .... | 3.7 | molten |
| 1.3 per cent nickel; remainder cadmium | 33.2 | 21.9 | .... | 6.5 | 1.8 |
| 3 per cent nickel; remainder cadmium | 47.5 | 28.4 | .... | 8.3 | 1.9 |
| High-lead alkaline earth | 27.4[a] | 15.7 | 10.1 | .... | .... |

[a] Temperature 68 F. (20 C.).

*Hardness Tests:*

(*a*) Rockwell hardness numbers were determined on the three alloys at 77 F. (25 C.) with the results shown below:

| | ROCKWELL HARDNESS NUMBERS | |
|---|---|---|
| COMPOSITION | "B" SCALE, ¼-IN. BALL, 100-KG. LOAD | "B" SCALE, ¼-IN. BALL, 60-KG. LOAD |
| 5.23 per cent copper; 6.2 per cent antimony; remainder tin | 51 | 86 |
| 1.35 per cent nickel; remainder cadmium | 65 | 92 |
| 3 per cent nickel; remainder cadmium | 84 | 103 |

(*b*) Brinell hardness numbers were determined on the three alloys as shown in Table II. Tests were made by keeping the full load on the specimen for 30 seconds. Values are the mean of at least three readings. A 10-mm. ball was used throughout.

(*c*) Microhardness values were made on the constituents of the tin-base and cadmium-base alloys at room temperature, using the Bierbaum microcharacter. For comparison the hardness of an ordinary steel shaft (cold-rolled) is included:

| | MICROHARDNESS NUMBERS |
|---|---|
| Tin-base babbitt alloy: | |
| hard constituent | 204 |
| matrix | 61 |
| Cadmium-base alloy: | |
| hard constituent | 260 |
| matrix | 55 |
| Steel shaft | 473 |

*Service Tests:*

After the physical tests on these alloys had progressed far enough to indicate a marked superiority in properties of the cadmium alloy over the tin alloy, several practical tests were made to check the theoretical findings. Bearings of a composition approximating 1.35 per cent nickel, remainder cadmium, were used in all tests.

(*a*) Light slow-speed duty tin babbitt in a ventilator fan was replaced by the cadmium bearing alloy. These bearings are still in service after having run an average of 60 hours per week since September, 1932.

(*b*) High-speed heavy-duty tin babbitt bearings in a pulverizing mill failed and were replaced by the cadmium bearing alloy. After these bearings were "worked in" they ran without further attention and at a somewhat lower temperature than the tin babbitt used previously.

(*c*) Automotive duty: The cadmium-base bearing alloy was further tested in a 1931 motor of popular make, using it to replace the two end main bearings and Nos. 2, 4 and 6 connecting-rod bearings. The center main bearing and Nos. 1, 3 and 5 connecting-rod bearings were those originally supplied in the motor by the manufacturer. The motor was mounted on a concrete block. After running a few hours at a low speed to "work in" the new bearings, it was coupled to an electric generator which could be given a variable load through suitable resistances by varying the field voltage. Motor temperatures were maintained considerably above those found in normal motor car operation. At the end of a period equivalent to 29,000 miles (671 hours) of continuous operation, the motor was taken down for inspection. All bearings were found to be in first-class condition. After reassembling, the test was continued to an equivalent of 52,500 miles (1197 hours) when a shut-down was necessitated due to failure of the babbitt bearings. During the test the motor was run continuously except to change oil every 24 hours and to grind valves, replace spark plugs and to make other necessary repairs and adjustments. An average of 32.4 hp. was developed throughout the test at an average speed approximately 2300 r.p.m.

When the motor was taken down it was found that the center main bearing had fused. A considerable amount of babbitt alloy was found in the crankcase. Connecting-rod bearings of tin-base babbitt all showed evidence of temperatures above the softening point of the babbitt alloy. All cadmium bearing alloys were in first-class condition and could have been used again in the motor.

## DISCUSSION OF DATA

*Compression tests* show the superior strength of the cadmium bearing alloy over the tin-base babbitt, especially at elevated temperatures. This is to be expected in as much as the hardness of the former is greater and the melting point is about 140 F. (78 C.) higher.

*Tension tests* indicate that the tin-base and the cadmium-base alloys possess about the same degree of ductility.

*Creep tests* show the decided superiority of the cadmium-bearing alloy over the tin-base babbitt in supporting loads and yet the creep resistance of the cadmium bearing alloy is not sufficiently high to prevent alignment during the working-in period of a bearing. This was shown by the fact that tool marks disappeared on the bearing surfaces of the cadmium bearings installed in the automobile motor without any measurable change in bearing thickness. The grouping of the three points on the cadmium-nickel curve indicates a varying amount of cold work due to difference in thickness of the specimen. The order of magnitude, nevertheless, remains practically the same.

*Hardness tests* show that the cadmium-base bearing alloy is much harder than the tin-base babbitt especially at elevated temperatures. At room temperature the microhardness of the matrices of the two alloys are about equal. The hard constituent of the cadmium-base alloy is much harder than that of the hard constituent of the tin-base alloy. Nevertheless the hardness is still lower than the hardness of any steel shaft likely to be used with the alloy. In this connection should be mentioned the fact that oxidation of the cadmium alloy will not develop any constituent which has a greater hardness than that of the cadmium bearing alloy, whereas the excessive hardness of tin oxide developed when tin babbitt is overheated is appreciated and examples of its damage to steel shafts are well known.

*Service tests* showed that the cadmium bearing alloy was suitable for both light and heavy loads, both low and high speeds and variable loads such as found in automotive motors. The test using the automobile motor showed the ability of the cadmium bearing alloy to withstand higher temperatures than the tin bearing alloy.

*Properties of Cadmium-Nickel Bearing Alloy:*

Inasmuch as the properties of cadmium-nickel bearing alloys are not generally known the following data are included:

Matrix material: eutectic of cadmium and the compound $NiCd_7$, the alloy analyzing 0.25 per cent nickel.

Hard constituent: $NiCd_7$.

Lower Limit of melting range (melting point of eutectic: 604 F., 318 C.).

Bonds readily with iron, steel, ferrous alloys in general, brass and bronze. (No trouble was encountered in "tinning" the connecting rods with the mixture and then casting the bearing onto the tinned surface to form a good bond.)

Easily cast using essentially the technique used in casting tin babbitts.

Not easily oxidized. This alloy shows greater resistance to oxidation than any other cadmium alloy of the many that have been studied.

Hardness increases with the nickel content.

Properties of the binary cadmium-nickel alloy may be altered by the addition of small amounts of other elements.

## CONCLUSION

In comparing the physical properties of tin-base babbitts and cadmium-nickel bearing alloys, especially at elevated temperatures, the cadmium-nickel alloy shows a superiority over tin babbitts. The following conclusions are believed to be justified from the data submitted:

1. The compressive strength of the cadmium-nickel bearing alloy is greater at all temperatures investigated than the ordinary tin-base babbitt.

2. The softening point of the cadmium-nickel alloy is higher than that of any tin-base babbitts now in use.

3. The creep tests indicate a much higher order of creep resistance in the cadmium-nickel alloy than in the tin alloy. Practical tests nevertheless show sufficient creep to serve the purpose of alignment in the various installations.

4. Elongation and reduction of area measurements show the ductility of the tin babbitt to be about equal to that of the cadmium alloy.

5. The hardness of the cadmium-nickel alloy is greater at all temperatures studied than the hardness of the tin babbitt. However, microhardness measurements show that the hard constituent is not too hard to act as bearing contact for the softest steel shaft likely to be used.

6. The cadmium-nickel alloy unlike other cadmium alloys is capable of readily bonding itself to steel, as well as to brass, and bronze, the alloys likely to be used as backing for bearings. Actual operating tests in various installations indicate that the cadmium-nickel alloy can be used in many installations to advantage. Its greater strength indicates greater load-carrying capacity, and its higher melting point gives a better performance at temperatures now considered above normal for bearings.

# DISCUSSION

**Mr. H. K. Herschman**[1] (*presented in written form*).—The paper by Messrs. Swartz and Phillips is a welcome contribution on this subject, particularly as it furnishes data on cadmium alloys for bearing service, on which there is a dearth of information.

The authors make the following statement in their paper: "Since various methods of impact testing do not appear to be standardized for bearing metals, and *since these values are rather closely related to hardness tests*, etc..." Such conclusions are not substantiated by the data determined by Herschman and Basil.[2] Thus, in Fig. 8 of the paper referred to, the cadmium-zinc alloy showed higher impact values up to 100 C. than the other alloys tested, but its resistance to impact at 150 and 200 C. decreased to a figure below that for the other alloys. On the other hand, the cadmium-zinc alloy had higher hardness up to 200 C. than any of these alloys as shown in Fig. 9 of that paper. Referring to alloy No. 1 (a high tin, antimony, copper alloy) and the alkaline earth hardened lead of that investigation, in general their resistance to impact increased as the temperature was increased while their hardness values under these conditions decreased. In the light of Boegehold's statement[3] that main crankshaft and connecting rod bearings are now being subjected to shock of 2000 lb. per sq. in. projected area and to temperatures of 200 C., it would appear that the impact factor cannot be so easily ignored nor discounted. The determination of impact values for the cadmium-nickel alloys would, I believe, make a valuable addition to the excellent paper presented by the authors.

**Mr. W. H. Swanger.**[4]—I agree with Messrs. Swartz and Phillips that the impact test has not been standardized, particularly for bearing metals. In many cases, an impact test of a particular metal shows what the metal will do in the impact test rather than serving as a criterion of the service life. However, as Mr. Herschman has shown, the impact test may serve as a useful method for the comparison of the behavior of different bearing metals at various temperatures.

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[1] Associate Metallurgist, U. S. Bureau of Standards, Washington, D. C.

[2] H. K. Herschman and J. L. Basil, "Mechanical Properties of White-Metal Bearing Alloys at Different Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 536 (1932).

[3] A. L. Boegehold and J. B. Johnson, "Engineering Requirements in the Automotive Industry for Metals Operating at High Temperatures," Symposium on Effect of Temperature on the Properties of Metals, published jointly by the American Society for Testing Materials and The American Society of Mechanical Engineers, p. 169 (1931).

[4] Metallurgist, U. S. Bureau of Standards, Washington, D. C.

MESSRS. L. C. BLOMSTROM[1] and E. R. DARBY[2] (*presented in written form*).—The performance of the cadmium-base alloy as recorded by Messrs. Swartz and Phillips is very interesting to us, particularly as the Federal-Mogul Corp.—working through a fellowship at Battelle Memorial Institute—has developed a cadmium-base metal for the same purpose. Laboratory and engine tests covering a period of four years have demonstrated a number of interesting features.

The alloy, one of cadmium, zinc, and antimony, has a melting point substantially higher than the tin-base babbitts. Its strength, hardness, and resistance to pounding are greater at all temperatures, while its rate of wear, as shown by Amsler wear tests, is considerably lower than for any other bearing metal tested.

Rod and main bearing tests on this material in a 60-hp. motor run at full load with a speed of 2900 r.p.m. showed, after several hundred hours continuous running, a decided superiority over tin-base metals. A car fitted with rods lined with the cadmium-base alloy made the trip from Detroit to Los Angeles and return at an average speed of slightly over 50 miles per hr. The trip was made without any wearing-in period whatever being given to the rods. In spite of a very high compression ratio and high motor speed, the rods came through this continuous run in almost perfect condition.

In a dynamometer rod test on a 90-hp. six-cylinder motor fully loaded at 3200 r.p.m., 216 hr. running failed to show any damage to the lining metal. The same rods placed in another motor were given 100 hr. additional running at the end of which they were still in excellent condition.

Where failures in service tests have occurred the cause has been traced in most cases to either "off-analysis" material or condition of bond. In no instances have failures been noticed at moderate speeds.

It would, in our opinion, have been more interesting to engine builders had the tests run by Messrs. Swartz and Phillips been made at higher speeds and greater power output. The usual test in the automotive industry is a 100-hr. dynamometer run at full load and speed. Such a test frequently develops an order of performance entirely different from that secured by long-time relatively low-speed runs. Wiping or seizure, cracking and other forms of lining failures are quite generally related to continuous high speed, high load and high temperatures. Bearings may run indefinitely at 2000 r.p.m. but fail almost instantly at 4000 r.p.m. A speed equivalent to 43 miles per hr. is quite low to show up lining metal qualities.

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[1] Federal-Mogul Corp., Detroit, Mich.

[2] Metallurgist, Federal-Mogul Corp., Detroit, Mich.

Main bearings as a rule do not give any trouble whatever at low speeds, but if trouble should develop on a three-bearing crankshaft, it will appear first in the center bearing. We should like to ask Messrs. Swartz and Phillips if they have tried the cadmium-nickel alloy in the center main bearing.

The creep tests are interesting. Would the same order of performance obtain at higher temperatures? What, in the opinion of the authors, would the tin-base alloy show at say 350 F. (175 C.)? Would not this throw light on the value of the creep test as a measure of bearing qualities?

MR. H. W. GILLETT[1] (*presented in written form*).—Beside the interesting data provided by Swartz and Phillips on cadmium with a little nickel, and the cadmium-zinc-antimony alloy[2] developed for the Federal-Mogul Corp. referred to above by Blomstrom and Darby, work done at the National Physical Laboratory by Murphy[3] on a cadmium alloy with a little copper and magnesium has just been reported. Murphy concludes that this alloy makes a satisfactory bearing and one of good high-temperature properties.

The only report at all adverse to the cadmium "babbitts" is that of Herschman and Basil[4] before the Society last year, who found greater wear on the cadmium-zinc eutectic than on tin-base babbitt. This is readily explained, however, by their failure to introduce any element that would form hard particles in the eutectic matrix, thus violating the ancient but still honorable requirement of a bearing metal for a definitely duplex structure on a larger scale than that of the eutectic. By introduction of antimony and suitable balancing of composition, structures are obtained very closely resembling those of the standard babbitt compositions.

MR. SAM TOUR.[5]—I should just like to get into the record a reference to some related work. The Battelle Memorial Institute carried out research on six copper-base bearing alloys jointly for the Copper and Brass Research Corp. and certain bearing metal manufacturers. A considerable amount of that work was reported recently in a paper before the American Foundrymen's Association.[6] One particular connection with the work just reported here is in the type of impact testing developed by Battelle Memorial Institute to study the pounding out of bearings.

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[1] Director, Battelle Memorial Inst., Columbus, Ohio.

[2] U. S. Patent 1,864,240, June 21, 1932.

[3] A. J. Murphy, "Recent Developments in Bearing Metals," *The Metal Industry* (London), Vol. 42, June 9, 1933, p. 591.

[4] H. K. Herschman and J. L. Basil, "Mechanical Properties of White-Metal Bearing Alloys at Different Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 536 (1932).

[5] Vice-President, Lucius Pitkin, Inc., New York City.

[6] O. E. Harder and C. S. Cole, "A Study of Six Bearing Bronzes," *Preprint No. 33-13*, Am. Foundrymen's Assn. (1933).

MR. C. E. SWARTZ[1] (*authors' closure*).—The results presented by Messrs. Blomstrom and Darby are very interesting. The tests run by them on different motors are comprehensive and indicate a superiority of the cadmium-zinc alloy over the present automotive babbitts. We agree that our tests would be more conclusive if they had been run at a higher speed and load had such condition been attainable with the motor used.

The center main bearing was standard tin babbitt, and it not only softened but unquestionably fused. In fact, a considerable amount of babbitt was found in the crankcase at the completion of the test. All cadmium bearings were in first-class condition in spite of the fact that the cadmium main bearings were cast solid, while the tin-base bearing was steel backed.

While Blomstrom and Darby's comments do not give the temperatures attained during their various tests, it is probable that the temperature of our motor test was somewhat higher. When the tests were started, oil temperatures between 115 and 136 C. were maintained, but constant spark plug and valve trouble necessitated lowering the temperature to 100 to 110 C. for the duration of the test.

It is difficult to predict whether the creep strength ratios between the tin and cadmium alloys would remain the same at elevated temperatures, but we can safely predict a similar order of performance.

Mr. Herschman raises the question of impact testing. It is not unlikely that had the authors facilities for making impact tests, such tests would have been reported. After considering the question of impact tests, and communicating with others who had used impact tests on bearing alloys it was decided to omit them.

A paper discussing the microstructure of the alloys of cadmium and nickel has recently been presented before the American Institute of Mining and Metallurgical Engineers, which is of interest in connection with the present study.

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[1] Central Research Laboratory, American Smelting and Refining Co., Maurer, N. J.

# THE EFFECT OF THE ADDITION OF LEAD ON THE HARDNESS OF CERTAIN TIN-BASE BEARING ALLOYS AT ELEVATED TEMPERATURES

By J. N. Kenyon[1]

## Synopsis

Lead additions to three tin-base alloys in amounts of 3 to 4 per cent increase the hardness of the material for temperatures up to 100 C. (Figs. 3 to 8). No important falling off in hardness due to these additions is found for temperatures up to the melting point of the tin-lead eutectic (183 C.) (Figs. 9, 10, 11).

Hardness-temperature curves for tin-base bearing alloys are parabolic in form and when plotted to logarithmic scale predict with a fair accuracy the hardness of the material for higher temperatures (Fig. 12).

Greater accuracy is attained in the making of hardness determinations if a load is chosen that will not cause "cratering" of the Brinell impression. Hardness values obtained with loads of 250 and 500 kg. do not differ materially except for a range of Brinell impressions showing appreciable "cratering" under the higher load (Fig. 13).

The purpose of this investigation is to determine the effect of small additions of lead on the hardness of certain tin-base bearing alloys within the normal working temperatures of the material (up to 100 C.) and to determine the effect of temperatures upwards to about 200 C. on the hardest alloys of the three series.

The lead content of tin-base bearing alloys is limited to a relatively low figure by British and American practice because it is thought to be injurious **(21,22)**.[2] Due to the "tinning" practice and possibly to lead being contained in commercial tin and antimony, small amounts of it may be present in these bearing materials **(9,12,15)**. "Wiping" has been found to occur at 170 to 180 C. with a lead content of 0.34 per cent which is within the limit set by American practice **(12)**. The effect of *any* lead on the physical properties of these bearing metals must therefore be taken into consideration.

Heretofore no serious difficulty has been experienced with tin-base bearing alloys in automotive engines; the material as a whole has given excellent service. With recent trends toward increased horsepower and higher working temperatures, bearing failures, if not actually encountered, are being anticipated. Engine bearings are now being subjected to temperatures reaching upwards to 200 C., the temperature at which tin-base alloys have a Brinell hardness of less than five(2) (Fig. 12). It is being predicted that the babbitts, as a material for automotive engine bearings, will eventually be abandoned (2).

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[1] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 443.

## BRIEF SURVEY OF AVAILABLE LITERATURE

The following surveys of existing literature are significant in the study of the subject:

*1903.*—Clamer investigated genuine babbitt metal (tin, 88.9 per cent; antimony, 7.4 per cent; copper, 3.7 per cent) and reported that lead in small amounts made this alloy "harder, stiffer, more easily melted and superior in every way"(6).

*1916.*—Förg says that an addition of 5 per cent of lead to tin-base alloys gives little change in structure. When added in larger amounts a new constituent appears which is softer than the tin-antimony-copper eutectic and results in a decrease in ball hardness. The alloy showing the least segregation regardless of the pouring temperature is the following: tin, 84 per cent; antimony, 10 per cent; copper, 5 per cent; lead, 1 per cent (7,13).

*1919.*—Jones made a series of hardness-temperature tests on a tin-base alloy with lead additions up to 5 per cent. The results are most interesting in that a *per cent increase in hardness* due to lead is shown for higher temperatures as compared with the hardness at room temperature (17).

*1919.*—The Engineering Aircraft Service, U. S. Department of War, reports chemical analyses of tin-base bearing metals taken from the crankshafts and connecting rods of four Liberty motors. Satisfactory service was reported although lead was found to be in excess of the 0.35 per cent limit. The lead contents were 1.85, 0.18, 2.52 and 2.87 per cent (1).

*1921.*—Freeman and Woodward investigated the effect of lead on the yield point and compressive strength of a tin-base bearing alloy. They state: "The additions of amounts up to 5 per cent of lead to this babbitt seem to have no very appreciable effect on its mechanical properties in compression at room temperature or at 75 C. under the conditions of test used. The authors think, however, that these tests should not lead to an increase in the lead content tolerance in tin-base bearing metal specifications until much more work is done along this line and particularly to determine the possible effect of small percentages of lead on the resistance to repeated impact"(14).

*1922.*—Mundey, Bissett and Cartland investigated the physical properties of several commercial alloys. The opinion was expressed that tin-base alloys are not harmed with small amounts of lead providing the lead is in even distribution. No testing data were offered to substantiate the statement (20).

*1922.*—Priestley submitted data which indicated that lead gave a substantial increase in hardness to tin-base bearing alloys. On the other hand he found an increased brittleness as indicated by the tendency to crack at lower compressive loads. Small additions of lead are thought to be beneficial since a greater resistance is offered to rapid changes in loading (21).

*1928.*—Cowan reports minute shrinkage cavities in tin-base alloys containing lead while practically none are found in these alloys containing no lead. These cavities are attributed to the freezing out of minute and isolated globules of a tin-lead eutectic at a temperature (183 C.) which is considerably below the freezing point of the bulk of the material (8).

*1928.*—Ellis and Karelitz considered lead additions to tin-base bearing alloys of importance because these alloys are subject to contamination. Their investigation showed that lead additions up to 1 per cent increased the hardness of the material but that this effect is completely lost at temperatures above 55 C. (11).

*1929.*—Ellis and Karelitz found that a tin-base bearing alloy containing 0.34 per cent lead showed a definite tendency to "wipe" at 170 to 180 C. At these temperatures the tin-lead eutectic has become "mushy"(12).

*1930.*—At the Detroit Regional Meeting of the Society, Clair Upthegrove presented a paper on "Automobile Bearing Metals." He states that tin-base bearing alloys are limited to 0.35 per cent lead "due to the fact that with temperatures appreciably above atmosphere, the effect of lead is to soften the metal regardless of the percentage present." No testing data were presented to substantiate the statement (23).

*1931.*—At the Symposium on Effect of Temperature on the Properties of Metals held jointly by the A.S.M.E. and A.S.T.M., Boegehold and Johnson presented interesting data regarding the future use of babbitt metal in the automotive industry. They state that main crankshaft and connecting-rod bearings are now being subjected to shock loads of 2000 lb. per sq. in. projected area and to temperatures of 200 C. Rapid failure of these bearings is being prevented by a reduction in thickness of the babbitt metal (from $\frac{1}{16}$ in. to $\frac{1}{32}$ in.), by crankcase oil temperature regulators and by avoiding a ***high lead content.*** They say: "The trend to higher and higher loads due to increased engine power and reducing weight will soon make it necessary to use bearing materials with higher softening points than that possessed by the babbitts"(2).

Darby presented a paper on bearing metals at higher temperatures in which he states: "The introduction of any appreciable amount of lead to the tin-base metals causes a lowering of their melting points approximately 100 F. (38 C.) and of course affects their physical properties accordingly"(10).

The following summaries have an indirect bearing on the subject under consideration:

*1923.*—Kiyosi Ito made hardness tests on a tin-base alloy for temperature increments between −49 C. and 145 C. The results show a hardness-temperature graph parabolic in form (16).

*1924.*—Lieber found that the hardness of bearing materials decreased after 20 minutes application of load. A load period of at least three minutes was recommended (7,18).

## PREPARATION OF MATERIAL

The alloys selected for the investigation are given in Table I.

The hardeners were made up of carefully weighed amounts of commercially pure copper and antimony. The material was placed in a graphite crucible, covered with charcoal, slowly heated in a gas-air furnace to 600 to 650 F. (315 to 345 C.) and held at this temperature until the copper was thoroughly dissolved in the antimony.

When no solid particles could be detected with a graphite stirring rod, the melt was cast into an iron solder mold.

The babbitt metal was made up by melting tin in a clay crucible with the hardener placed in the bottom. The crucible was kept covered and held at a low temperature in an electric furnace until the hardener was thoroughly dissolved in the tin. The charge was then thoroughly stirred, $NH_4Cl$ sprinkled over the top and cast into an iron-solder molder.

The bars of babbitt metal were sheared, mixed and weighed with the correct lead additions after which each charge was slowly melted in a covered clay crucible in an electric furnace. The molten metal was thoroughly mixed by pouring back and forth into a preheated crucible, returned to the furnace and brought to the correct casting temperature. $NH_4Cl$ was sprinkled over the bath after which the charge was cast into 1 by 1 by $\frac{1}{2}$ in. chilled steel molds. Previous to casting, the molds were chilled in cold tap water, carefully dried and a square of fine tissue paper placed in the bottom. The paper retarded freezing of the first drop of metal to strike the steel and gave a smooth bottom surface to the specimen. The specimens were annealed for two hours in boiling water and slowly cooled. The bottom surfaces were turned off about $\frac{1}{32}$ in. for the Brinell hardness impression.

TABLE I.—ALLOYS USED IN INVESTIGATION.

| | ALLOY NO. 1 (NAVY BEARING: HARD) | ALLOY NO. 2 (A.S.T.M. ALLOY GRADE NO. 2[a]) | ALLOY NO. 3 (A.S.T.M. ALLOY GRADE NO. 1[a]) |
|---|---|---|---|
| Straits tin, per cent | 80.0 | 89.0 | 91.0 |
| Antimony, per cent | 15.0 | 7.5 | 4.5 |
| Copper, per cent | 5.0 | 3.5 | 4.5 |
| Lead, per cent | 0 to 14 by addition | | |
| Hardeners: | | | |
| Tin, per cent | .... | .... | 50 |
| Antimony, per cent | 75 | 75 | 25 |
| Copper, per cent | 25 | 35 | 25 |
| Pouring temperature | 700 F. (370 C.) | 650 F. (345 C.) | 650 F. (345 C.) |
| Mold temperature | 45 to 50 F. (7 to 10 C.) | 40 to 45 F. (4.5 to 7 C.) | 40 to 45 F. (4.5 to 7 C.) |
| Steel molds | 1 by 1 by $\frac{1}{2}$ in.; wall thickness $\frac{3}{4}$ in. | | |

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[a] Standard Specifications for White Metal Bearing Alloys (known commercially as "Babbitt Metal") (B 23 – 26), 1930 Book of A.S.T.M. Standards, Part I, p. 707.

## DESIGN OF HEATING APPARATUS

An asbestos-lined pan was enclosed in a wooden box and fitted with an 8 by 4 by $\frac{1}{2}$-in. steel plate supported longitudinally on $\frac{1}{2}$-in.

square steel bars. Spiral heating coils, strung with earthen insulators, rested on the bottom of the container, under and around the steel plate.

A second pan, fitted with an 8 by 4 by $\frac{1}{4}$ in. steel plate, rested directly on the first steel plate and was partly filled with a transparent mineral oil of high flash point. The outer container was nearly filled with a heavy, high flash point oil thus totally immersing the heating coils and surrounding the inner container. (See Figs. 1 and 2.)

The novel feature of this design is a heavy mass of steel in direct metallic contact with the specimens and the whole insulated in a bath of low conductivity oil. No serious fluctuation in temperature occurred during testing and a whole series of specimens were tested together under nearly identical temperature conditions. Another feature of the design was a light triangular guide held in spring contact with the spindle of the Brinell testing machine. By means of this guide the specimens were brought in correct position for the Brinell impression without appreciable effort on the part of the operator.

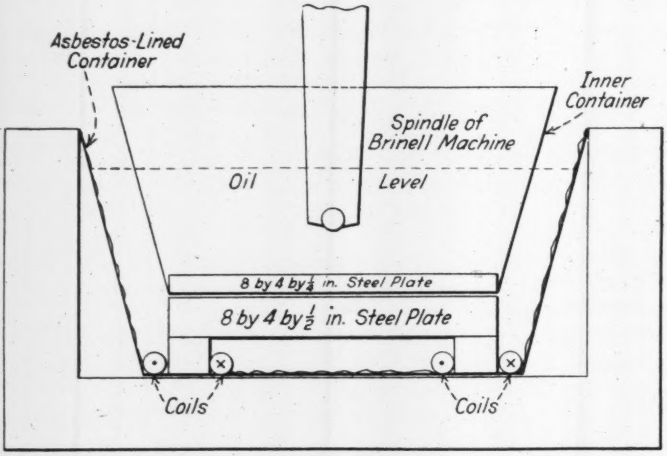

Asbestos-Lined Container
Inner Container
Spindle of Brinell Machine
Oil
Level
8 by 4 by $\frac{1}{4}$ in. Steel Plate
8 by 4 by $\frac{1}{2}$ in. Steel Plate
Coils
Coils

FIG. 1.—Diagrammatic Sketch of Heating Apparatus.

## TESTING PROCEDURE

Brinell hardness testing machines of the hydraulic type have a small frictional factor that should be given consideration for loads under 500 kg. The machine used in the investigation was tested with a manometer connected with the pressure chamber and found to have negligible friction. Considerable care must also be exercised with softer metals at higher temperatures since any impact effect in applying the load may result in appreciable error.

It is accepted practice to use a 500-kg. load applied for 30 seconds in making hardness determinations on non-ferrous metals. An exception to this must be made at higher temperatures due to the excessive "cratering" effect produced by the Brinell ball. The Brinell hardness number is expressed by the relation:

$$\text{B.H.N.} = \frac{P}{A} = \frac{\text{Total Pressure}}{\text{Area of Curved Impression}}$$

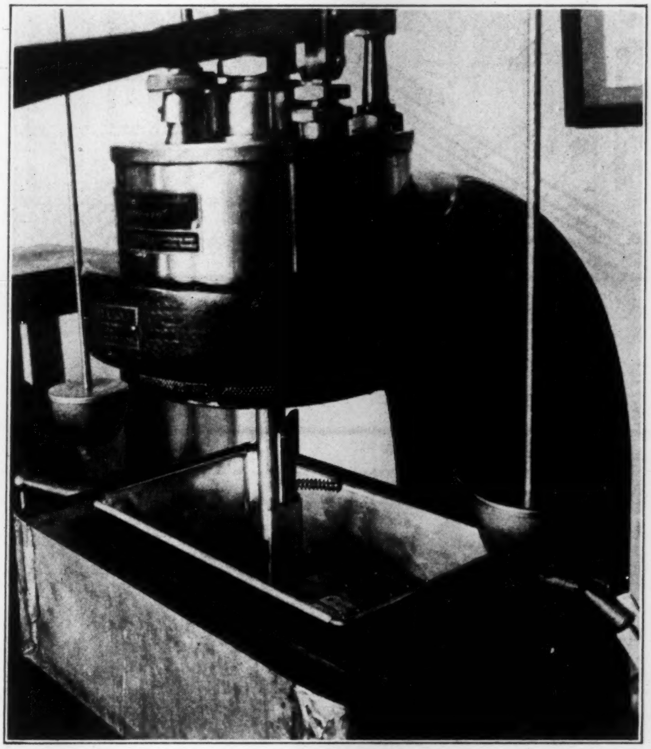
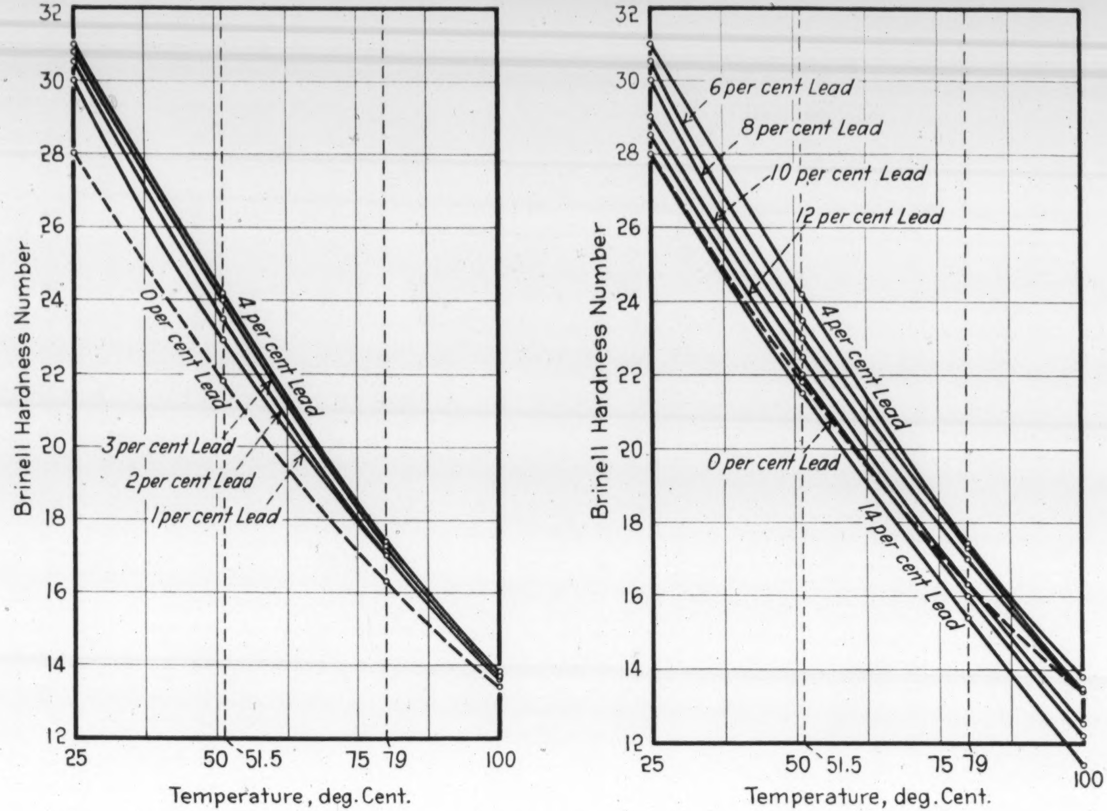


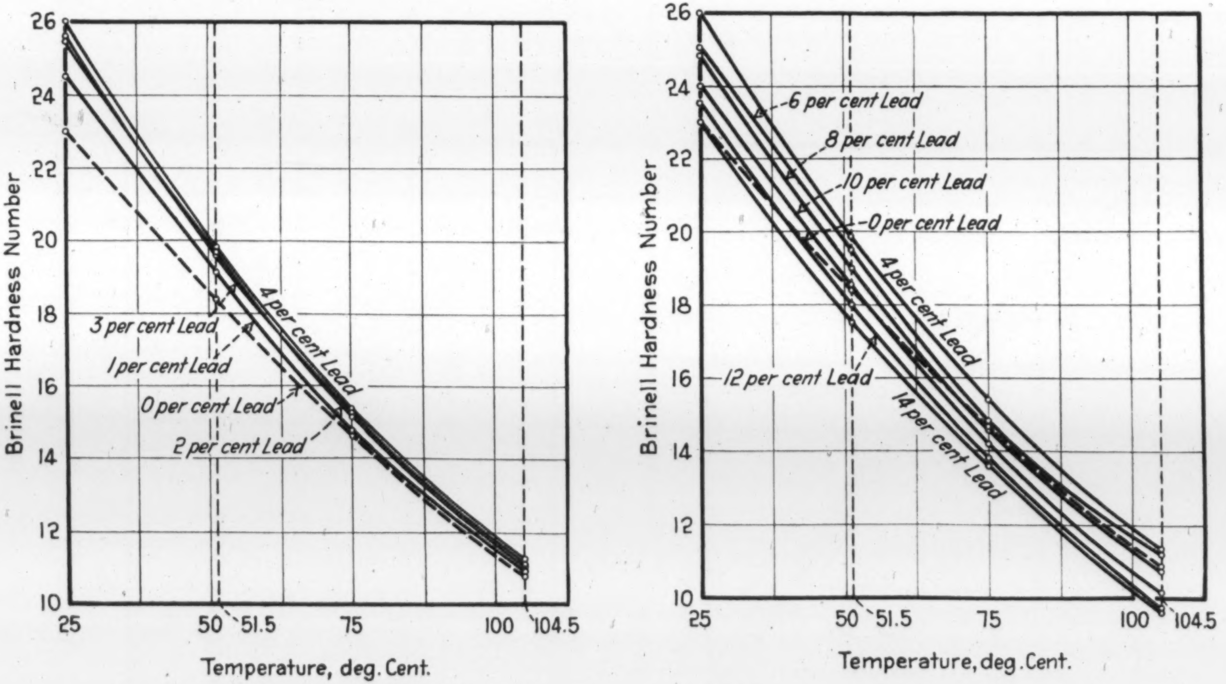
FIG. 2.—Testing Apparatus.

where "Different values of $P$ actually give different hardness numbers; probably a hardness number based on $P^n$ instead of $P$, where $n$ is a constant for a given material. ."(20). In order to determine the probable error involved, a series of hardness tests were made at different temperatures with loads of 250 and 500 kg., respectively. The hardness impressions for each load were made on diagonally opposite corners of the same specimen and the values for ten specimens averaged. No material variation in hardness values was found except for a range of impressions showing an appreciable cratering effect.



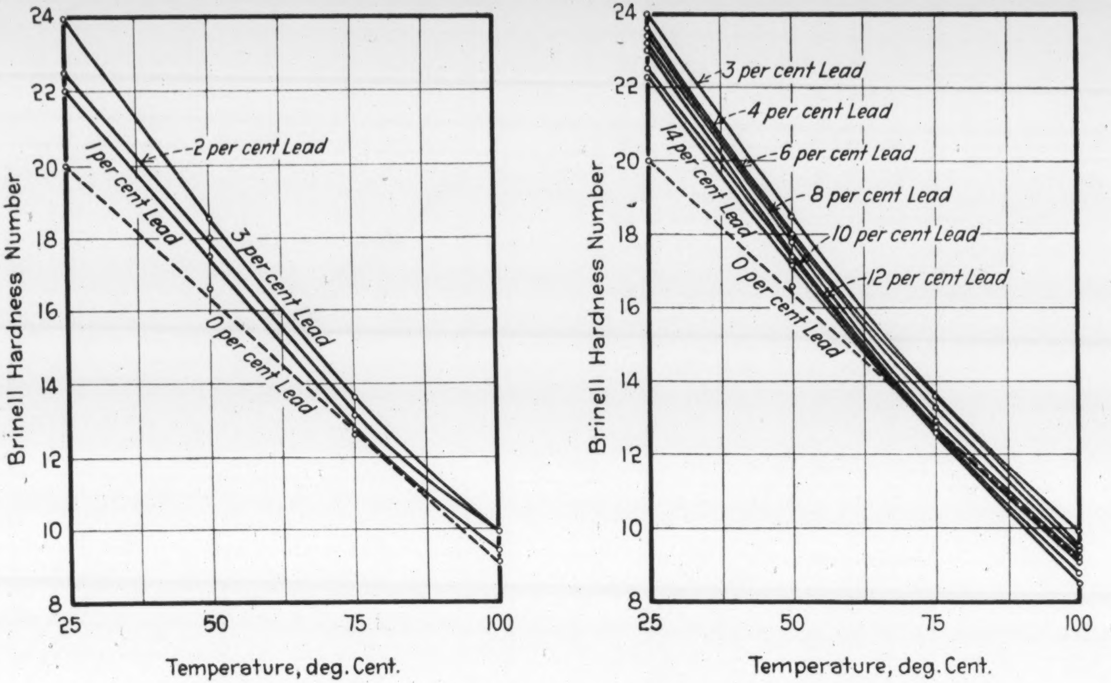
FIGS. 3 and 4.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 1.

Brinell hardness test using 10-mm. ball, 250-kg. load, 30 seconds. Each value represents the average of four deerminations.



FIGS. 5 and 6.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 2.

Brinell hardness test using 10-mm. ball, 250-kg. load, 30 seconds. Each value represents the average of four determinations.



FIGS. 7 and 8.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 3.

Brinell hardness test using 10-mm. ball, 250-kg. load, 30 seconds. Each value represents the average of four determinations.

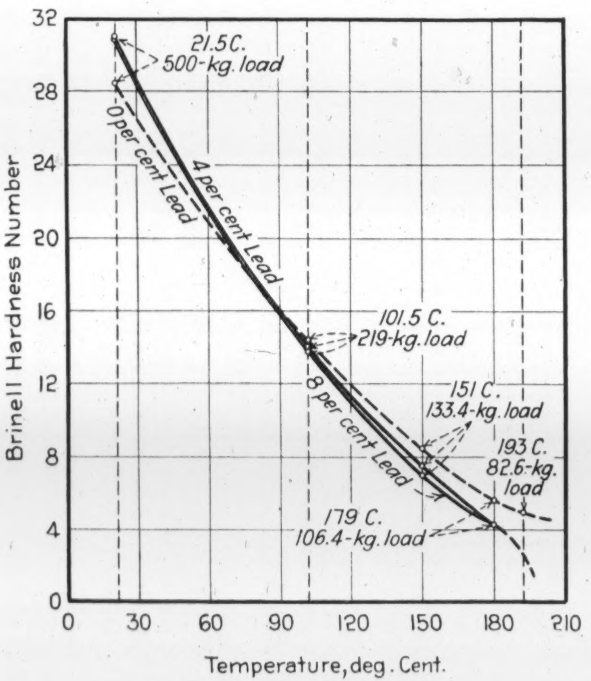


FIG. 9.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 1 Loaded to Give About 4.50-mm. Impression.
**Brinell hardness test using 10-mm. ball, 30 seconds. Each value represents the average of four determinations.**

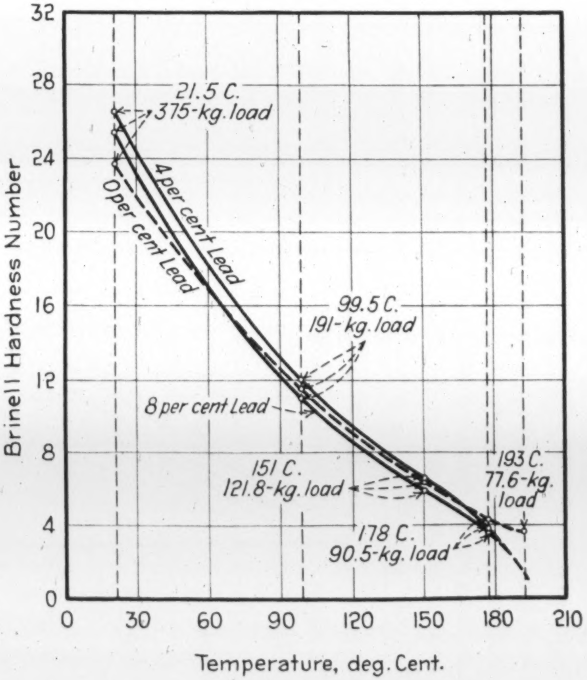


FIG. 10.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 2 Loaded to Give About 4.50-mm. Impression.
**Brinell hardness test using 10-mm. ball, 30 seconds. Each value represents the average of four determinations.**

The specimens were placed in the oil bath in close contact and in numerical order. The temperature was raised to the desired increment and held approximately constant for one hour, after which the hardness impressions were made. A thermometer fitted in a blank was used to record temperatures.

Four specimens were cast for each lead addition and one hardness impression made on each for a given temperature increment. The diameters of these four impressions were averaged and the results expressed as a single Brinell hardness number. In other words, each specimen has Brinell hardness impressions for four temperature increments: 25, 50, 75, and 100 C.

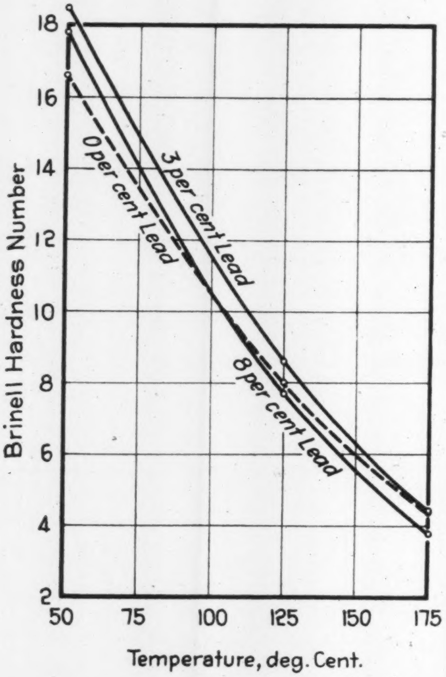


FIG. 11.—Effect of Adding Lead on the Brinell Hardness of Alloy No. 3 Loaded to Give About 4.50-mm. Impression.

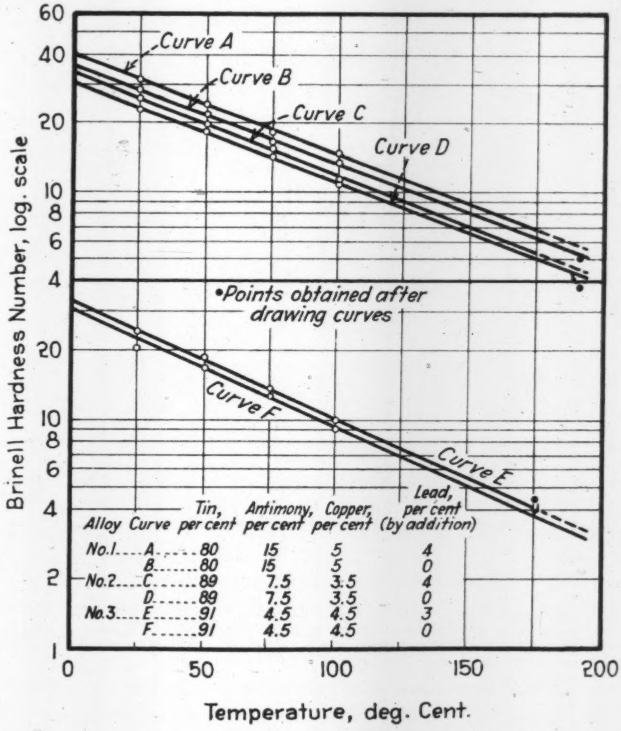
Brinell hardness test using 10-mm. ball, 30 seconds. Each value represents the average of four determinations.

## SUMMARY OF RESULTS

The data obtained in this investigation are submitted in the form of curves (Figs. 3 to 13).

*Figures 3 to 8:*

The addition of 4 per cent of lead to alloys Nos. 1 and 2 (Figs. 3 to 6) and of 3 per cent of lead to alloy No. 3 (Figs. 7 and 8) gave the greatest increase in hardness for all temperatures up to 100 C. The addition of 8 per cent of lead gave an increased hardness at room emperature but none at 100 C.



| Alloy | Curve | Tin, per cent | Antimony, per cent | Copper, per cent | Lead, per cent (by addition) |
|---|---|---|---|---|---|
| No. 1 | A | 80 | 15 | 5 | 4 |
| | B | 80 | 15 | 5 | 0 |
| No. 2 | C | 89 | 7.5 | 3.5 | 4 |
| | D | 89 | 7.5 | 3.5 | 0 |
| No. 3 | E | 91 | 4.5 | 4.5 | 3 |
| | F | 91 | 4.5 | 4.5 | 0 |

FIG. 12.—Brinell Hardness Tests Plotted to Logarithmic Scale.

*Figures 9 to 11:*

No important falling off in hardness due to the addition of 3, 4 and 8 per cent of lead was found for temperatures up to 183 C. Above this temperature the specimens containing lead crumbled under the applied load.

*Figure 12:*

The hardness values of three bearing alloys for temperatures up to 100 C. are plotted to logarithmic scale and the straight-line relations extrapolated in order to predict the probable hardness for

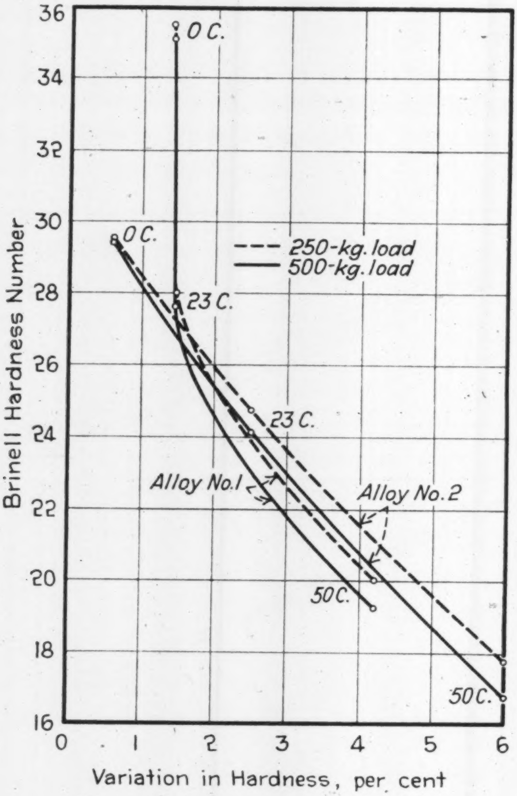


FIG. 13.—Showing the Variation in Hardness Numbers Obtained with Loads of 500 kg. and 250 kg.

Each value represents the average of 20 determinations.

higher temperatures. The following table is a summary of hardness values taken from these curves and later determined experimentally:

| Lead, per cent | Method of Securing Brinell Hardness | Alloy No. 1 | | | Alloy No. 2 | | | Alloy No. 3 | |
|---|---|---|---|---|---|---|---|---|---|
| | | 151 C. | 179 C. | 193 C. | 151 C. | 178 C. | 193 C. | 125 C. | 175 C. |
| 0.... | Extrapolated................ | 8.0 | 5.9 | 5.2 | 6.3 | 4.8 | 4.5 | 6.9 | 3.8 |
| | Determined................ | 8.4 | 5.6 | 5.0 | 6.4 | 4.3 | 3.7 | 8.0 | 4.4 |
| 3 ... | Extrapolated................ | ... | ... | ... | ... | ... | ... | 7.5 | 4.1 |
| | Determined................ | ... | ... | ... | ... | ... | ... | 8.6 | 4.4 |
| 4 ... | Extrapolated................ | 8.7 | 6.4 | ... | 6.9 | 5.1 | ... | ... | ... |
| | Determined................ | 7.5 | 4.3 | [a] | 6.6 | 3.8 | [a] | ... | ... |

[a] At this temperature the specimens containing lead crumbled under the applied load.

*Figure 13:*

The variation in hardness values obtained with loads of 250 and 500 kg. is shown.

The hardness values obtained with the two loads varied about 1 per cent for impressions up to 5 mm. in diameter; with an impression of 6 mm. and a tendency to crater the difference in hardness was found to be about 6 per cent.

Hardness determinations at temperatures above 100 C. were made with loads that gave about 4.5 mm. diameter impressions. The results checked with reasonable accuracy the values obtained by extrapolation from Fig. 12.

*Acknowledgments.*—The author gratefully acknowledges indebtedness to Prof. William Campbell for his invaluable advice in the making of this investigation. Appreciation is due to the Civil Engineering Testing Laboratories of Columbia University for the unlimited use of equipment, and to Paul H. Ripley for the preparation of curves.

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# DISCUSSION

MR. H. K. HERSCHMAN[1] (*presented in written form*).—Mr. Kenyon's paper furnishes some interesting and desirable information regarding the effect of varying percentages of lead on the hardness of tin-base bearing alloys. Some caution is, however, necessary in applying such data, for hardness values alone may not be depended upon to indicate the suitability of a bearing alloy for service.

There are indeed many metallurgical factors which must be considered when making a laboratory study for the selection of an alloy for bearing service, and these can be determined only by considering all of the mechanical test data significant in the service of a bearing. Thus, the author quotes in his paper results of work by Cowan[2] who reported that minute shrinkage cavities were found in tin-base alloys containing lead and that these are supposed to result in an embrittlement commonly associated with tin-base alloys containing lead. These findings would obviously suggest the advisability of "single-blow" or "repeated-blow" impact tests or both to determine the impact values of tin-base alloys containing lead. Furthermore, brittleness would be expected to affect adversely the wear-resistance properties of the alloy.

While the author indicated in his conclusions that Figs. 3, 5 and 7 show that certain lead additions increase the hardness up to 100 C., it would appear that the magnitude of the differences between the alloys with and without lead, at 75 and 100 C., are negligible.

Does the author consider that the same differences in hardness would have obtained had the specimens been cast in preheated molds such as is the usual commercial practice?

MR. J. N. KENYON.[3]—It is realized that hardness alone is not the whole story when it comes to determining the suitability of a bearing material for service. The author has under way another study to determine the effect of lead on the fatigue limit of tin-base bearing alloys, using an R. R. Moore fatigue machine for the purpose. While there are as yet no positive results to offer, typical fatigue fractures are being obtained. It is hoped that this investigation will bring out definitely the effect of the "minute shrinkage cavities" in tin-base bearing alloys containing lead as reported by Mr. W. A. Cowan.(8)[1]

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[1] Associate Metallurgist, U. S. Bureau of Standards, Washington, D. C.

[2] W. A. Cowan, "Minute Shrinkage Cavities in Some Cast Alloys of Heterogeneous Structure," *Journal*, Inst. Metals, Vol. 38, p. 53 (1928).

[3] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.

The addition of lead shows a small but definite *percentage* increase in hardness at higher temperatures as shown by the following table:

| ALLOY | TEMPERATURE, DEG. CENT. | BRINELL HARDNESS NUMBER[a] 0 PER CENT LEAD | 3 PER CENT LEAD | 4 PER CENT LEAD | PERCENTAGE GAIN |
|---|---|---|---|---|---|
| Tin 80, antimony 15, copper 5 | 25 | 28.0 | .... | 31.0 | 10.7 |
| | 100 | 13.4 | .... | 13.8 | 3.0 |
| Tin 89, antimony 7.5, copper 3.5 | 25 | 23.0 | .... | 26.0 | 13.0 |
| | 100 | 10.8 | .... | 11.3 | 4.6 |
| Tin 91, antimony 4.5, copper 4.5 | 25 | 20.0 | 24.0 | .... | 20.0 |
| | 100 | 9.1 | 9.9 | .... | 8.8 |

[a] See Figs. 3, 5 and 7 of the paper.

This increase in hardness is important in that the American practice is strictly to limit lead in tin-base bearing alloy because it is supposed to have a softening effect.

(*Author's closure, by letter*).—The question was raised by Mr. Herschman as to whether the same temperature-hardness relations would have obtained had the molds been preheated according to the usual commercial practice. Accordingly a series of tests were made on material cast into molds heated to 175 C., the temperature at which the tin-lead eutectic is "mushy." (12)[1] Other testing conditions

TABLE I.—BRINELL HARDNESS VALUES ON SPECIMENS CAST IN CHILLED AND IN HEATED MOLDS.

| Lead, per cent | Tested at 25.5 C. | | Tested at 76.5 C. | | Tested at 126.5 C. | | Tested at 176.5 C. | |
|---|---|---|---|---|---|---|---|---|
| | Chilled Mold[a] | Heated Mold | Chilled Mold[a] | Heated Mold | Chilled Mold[a] | Heated Mold | Chilled Mold[a] | Heated Mold |
| ALLOY: TIN 80.0 PER CENT, ANTIMONY 15.0 PER CENT, COPPER 5.0 PER CENT | | | | | | | | |
| 0 | 27.4 | 29.7 | 18.3 | 18.1 | 11.1 | 11.4 | 6.0 | 6.3 |
| 4 | 29.6 | 32.3 | 18.7 | 18.7 | 10.4 | 11.1 | 4.7 | 5.2 |
| 8 | 29.4 | 30.4 | 18.4 | 17.5 | 10.0 | 10.1 | 4.6 | 4.3 |
| ALLOY: TIN 89.0 PER CENT, ANTIMONY 7.5 PER CENT, COPPER 3.5 PER CENT | | | | | | | | |
| 0 | 22.8 | 24.1 | 14.4 | 15.0 | 8.6 | 8.8 | 4.4 | 4.7 |
| 4 | 25.6 | 26.6 | 15.4 | 15.3 | 8.9 | 8.8 | 4.0 | 4.2 |
| 8 | 24.5 | 23.5 | 14.3 | 14.6 | 8.0 | 8.1 | 3.6 | 3.7 |
| ALLOY: TIN 91.0 PER CENT, ANTIMONY 4.5 PER CENT, COPPER 4.5 PER CENT | | | | | | | | |
| 0 | 19.9 | 19.9 | 13.3 | 12.5 | 7.9 | 7.4 | 4.3 | 4.1 |
| 3 | 23.9 | 23.4 | 14.7 | 13.4 | 8.5 | 8.1 | 4.4 | 4.1 |
| 8 | 23.0 | 22.3 | 13.9 | 13.4 | 7.6 | 8.1 | 3.7 | 4.0 |

[a] Hardness values taken from Figs. 9, 10, 11 of the paper.

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[1] Refers to references appended to the paper, see p. 443.

were held constant. The results are compared with values taken from Figs. 9, 10 and 11 of the paper as shown in the accompanying Table I.

The above comparative results would seem to indicate that mold temperature has little effect on the temperature-hardness relations except in the case of the alloys with higher antimony content and at room temperature.

Mr. W. A. Cowan suggested in conversation that it would be advisable to investigate the effect of lead additions in amounts of less than 1 per cent. Tests were made on a tin-base alloy with these small lead additions and at a temperature well above the melting point of the tin-lead eutectic. It was found that the specimens crumbled under the applied load when lead was present in excess at 2 per cent. This phenomenon would suggest the presence of a liquid phase. The results on an alloy of tin 89 per cent, antimony 7.5 per cent, and copper 3.5 per cent are given below. The tests were made at 201.5 C. (10-mm. ball; 70-kg. load; 30 sec.):

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Lead, per cent | 0.00 | 0.33 | 0.66 | 1.00 | 1.33 | 1.66 | 2.00 | 2.33 | 2.66 | 3.00 |
| Brinell hardness | 3.5 | 3.4 | 3.4 | 3.4 | 3.3 | 3.3 | 3.1 | 2.9[a] | 2.5[a] | 2.2[a] |

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[a] Brinell hardness *approximate only* since the material showed a tendency to crumble under the load.

# THE EFFECT OF SULFUR AND IRON ON THE PHYSICAL PROPERTIES OF CAST RED BRASS (85 Cu, 5 Sn, 5 Zn, 5 Pb)[1]

By H. B. Gardner[2] and C. M. Saeger, Jr.[3]

## Synopsis

This paper is a progress report on the investigation sponsored by the Non-Ferrous Ingot Metal Institute at the U. S. Bureau of Standards. The object of this investigation is the collection of data to be used in the further simplification of the number of typical compositions of copper-base ingot metals. In the present paper all work has been restricted to the alloy, made of remelted metal, having the nominal composition of 85 per cent copper and 5 per cent each of zinc, tin and lead.

Three distinct types of test bars were cast at different pouring temperatures. In the study of the effect of sulfur, heats were cast at five pouring temperatures from 1900 to 2300 F. (1040 to 1260 C.) with additions ranging from 0.025 to 0.10 per cent sulfur. In the study of the effect of iron, heats were cast at the pouring temperatures of 1950, 2100, and 2250 F. (1065, 1150, and 1230 C.), with the additions ranging from 0.1 to 0.60 per cent iron. Tensile strength, Brinell hardness, electrical resistivity and density were determined on each bar and the results were compared with those for remelted metal. Casting at a high temperature showed a greater influence on the lowering of the physical properties of this brass composition than has the addition of sulfur. The addition of iron showed an improvement in all physical properties of this brass composition with the exception of the electrical resistivity. The running qualities of the alloy were improved and the shrinkage was unchanged by the addition of sulfur and iron.

## Introduction

In 1929 a special committee of the Society made a survey of commercial copper-base alloys. Upon the recommendation of this Society, the Non-Ferrous Ingot Metal Institute sponsored an investigation at the U. S. Bureau of Standards of the typical alloys of this group. Work began in June, 1930, under the research-associate plan. The first alloy studied was brass containing 85 per cent copper and 5 per cent each of lead, tin and zinc. A paper presented before this Society[4] in 1932 contained the results obtained on the effect of the type of test bar and of the pouring temperature upon the physical properties of this brass when made from virgin metal and from remelted metal.

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[1] Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate of the Non-Ferrous Ingot Metal Institute at the U. S. Bureau of Standards, Washington, D. C.

[3] Physicist, U. S. Bureau of Standards, Washington, D. C.

[4] H. B. Gardner and C. M. Saeger, Jr., "Factors Affecting the Physical Properties of Cast Red Brass (85 Cu, 5 Zn, 5 Sn, 5 Pb)," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 517 (1932).

The next phase of the investigation was the effect of impurities on the physical properties of the alloy (made from remelted metal) and of permissible limits of specific impurities. The general belief has been that sulfur and iron have an injurious effect upon physical properties of the non-ferrous alloys, although very few quantitative data have been published. In the survey made in 1929, it was found that in the group of which this alloy is representative, approximately only 10 per cent permitted more than 0.05 per cent sulfur or 0.3 per cent iron. In the present paper are reported the effects of sulfur and iron as separate impurities in this alloy.

In carrying out this phase of the work a reduction was made in the number of types of test bars. Those used were the immersion-crucible bar, the end-gate bar, both "cast-to-size" and "machined-to-size," and the $\frac{1}{4}$-in. web fin-gate bar. These were cast from heats of remelted brass to which from 0.025 to 0.10 per cent of sulfur had been added. They were cast at pouring temperatures from 1900 to 2300 F. (1040 to 1260 C.). To curtail expense and to expedite the investigation, only three pouring temperatures were used in casting the bars to which iron additions had been made. The physical properties of remelted metal given in the diagrams for iron for the pouring temperatures of 1950 and 2250 F. (1065 and 1230 C.) were the averages respectively of the values for the physical properties of remelted metal poured at 1900 and 2000 F. (1040 and 1095 C.), and at 2200 and 2300 F. (1205 and 1260 C.), which values were shown in the paper presented in 1932.[1]

The following physical properties were studied in sequence on each bar: electrical resistivity, tensile properties, density and Brinell hardness. In addition, the shrinkage of the metal in the liquid and the solid state and the running properties when cast in a green-sand mold were determined.

## MATERIALS AND METHODS OF CASTING

*Materials:*

Virgin metals of a grade similar to those reported in 1932 were used. "Stick sulfur" was added, and for the addition of iron, a copper-iron "hardener," containing 52 per cent of iron, was prepared from virgin copper and commercial open-hearth ingot iron.

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[1] *Loc. cit.*

*Molding, Melting and Pouring:*

A stock was prepared from virgin metal in a high-frequency induction furnace of the "lift-coil" type and was cast into pigs. The stock was melted in 90-lb. heats in double-wall clay-graphite crucibles. Each melt was poured at a definite pouring temperature after it had been heated to a temperature 100 F. (56 C.) higher. The double-wall crucible was necessary to minimize the temperature drop during pouring. The difference in pouring temperature between the first and last of a series of bars was about 16 F. (9 C.) at 1900 F. (1040 C.) and about 30 F. (16 C.) at 2300 F. (1260 C.).

Green-sand molds were prepared from grade 00 Albany sand. A permeability number of 12 to 16, a compressive strength of 5 to 7 lb. per sq. in. (American Foundrymen's Association units) and a moisture content of 6 to 6½ per cent were maintained.

The sulfur additions were made by placing the amount of stick sulfur, found by preliminary melts and analyses to be necessary, in an inverted, ventilated graphite crucible which was submerged in the molten metal until all agitation due to the reaction of the sulfur had ceased. The copper-iron hardener was added just before the charge was melted down. This gave a uniform mixture in the crucible. Subsequent analyses of the alloys showed that in all cases the contents of sulfur and iron were within 5 per cent of those specified.

*Types of Test Bars:*

Sand-cast bars, a chilled ingot, and two ingot immersion samples were obtained from each heat. Dimensioned drawings of each of these types of bars were shown in the previous paper.[1] The chilled ingots were not tested, but were stored for possible future tests to supplement the data given in this paper.

## RESULTS AND DISCUSSION

*Tensile Strength:*

The tensile properties were determined with an Amsler universal testing machine of 50,000-lb. capacity, the load being applied uniformly for all bars at a rate of 0.1 in. per min. The standard test specimen used was 0.505 in. in diameter, with threaded ends and a 2-in. gage length. In general, the reported results are the averages of duplicate determinations, usually on bars from one heat. In most cases the tensile strengths of the duplicate bars agreed within about 1000 lb. per sq. in. (about ±3 per cent), although there were a few larger variations. In such cases the higher values were accepted as being more authentic. Differences of less than 1000 lb. per sq. in. in the diagrams are probably not significant.

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[1] *Loc. cit.*

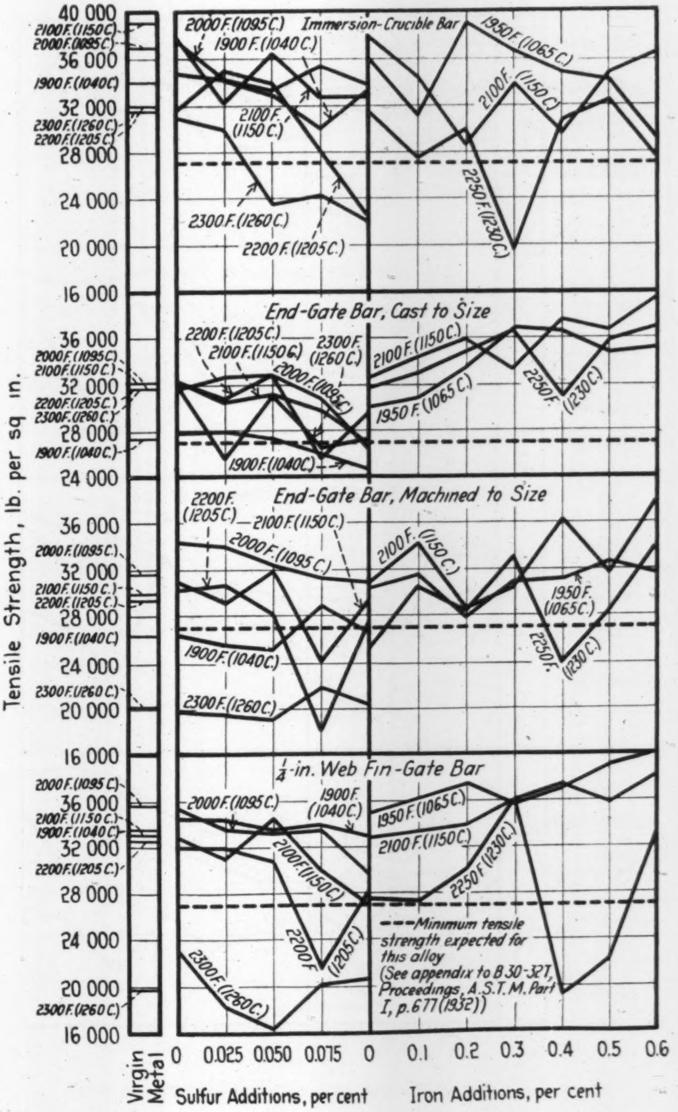

FIG. 1.—Effect of Sulfur and Iron upon the Tensile Strength.

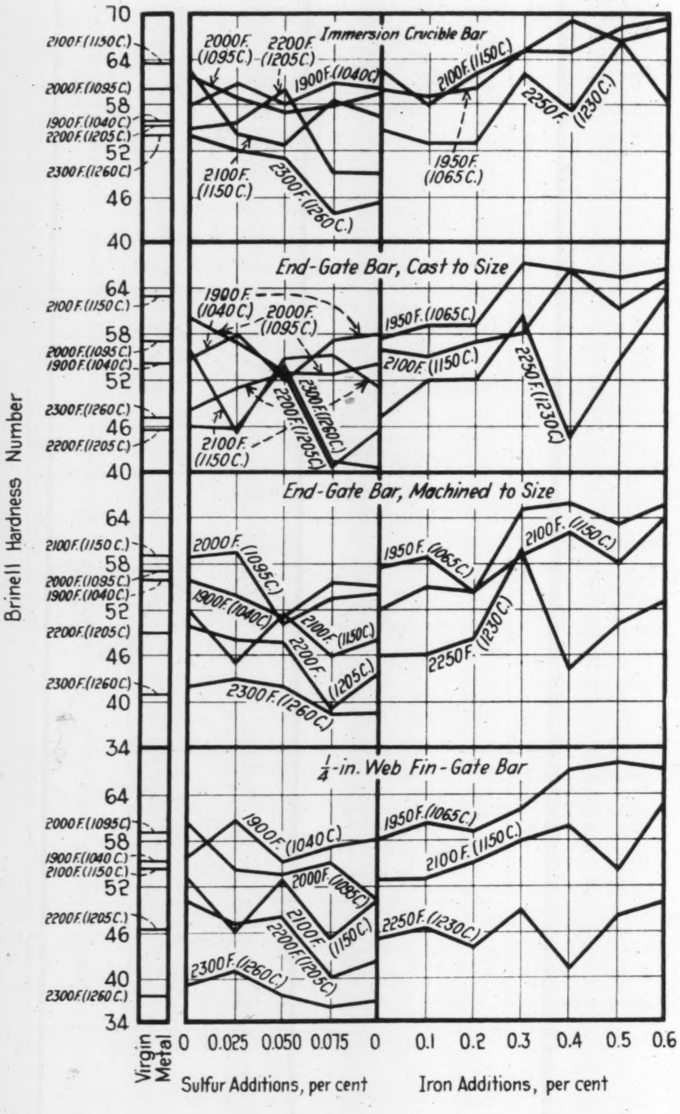

FIG. 2.—Effect of Sulfur and Iron upon the Brinell Hardness.

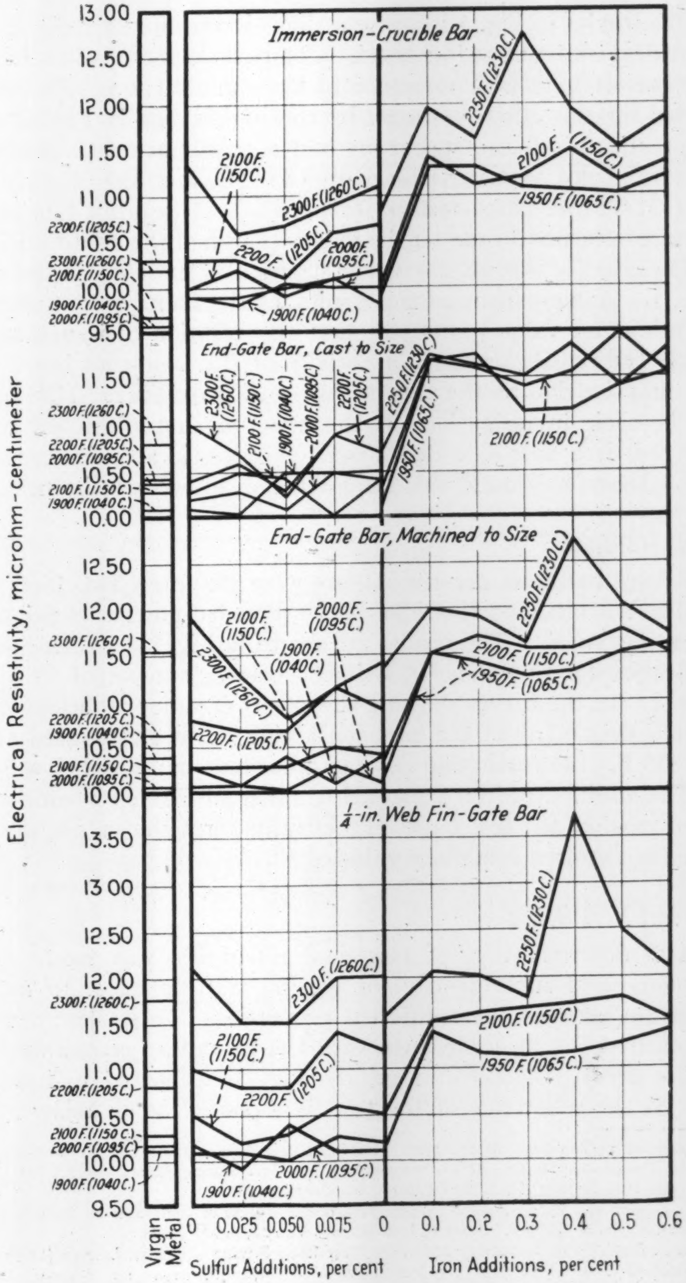

FIG. 3.—Effect of Sulfur and Iron upon the Electrical Resistivity.

The results in Fig. 1 indicate a slight lowering in tensile strength as the sulfur content was increased. Comparison of the values for these four types of bars with reference to "the minimum tensile strength expected for this alloy"[1] (shown by the broken line in Fig. 1) shows that practically all the specimens had a tensile strength above this minimum except some of those poured at 2200 and 2300 F. (1205 and 1260 C.). From these results it appears that pouring temperature had more influence on the tensile values than had the sulfur additions.

The effect of iron on the tensile strength of this alloy is also shown in Fig. 1. A comparison of the results for the four types of bars with the "minimum value" indicates that the addition of iron tended to increase the tensile strength, especially of the sand-cast bars. This effect may be due to the iron acting as a deoxidizer. Hanson and Ford in their investigation on the effect of impurities on copper[2] found that iron acts as a deoxidizer for copper in an oxidizing atmosphere. Dews[3] has found this same reaction to occur in bronzes.

### *Brinell Hardness:*

Brinell hardness determinations were made on flat faces, $\frac{5}{8}$ in in width, machined on the sides of the threaded portion of the broken tension specimens. The results are shown in Fig. 2. In the Society's specification for this alloy,[1] a Brinell hardness number of 50 to 60 is required. In the curves showing the effects of sulfur additions, it may be noted that many of the bars containing sulfur and poured at 2200 and 2300 F. (1205 and 1260 C.) had a hardness number below 50.

The addition of iron appeared to raise the Brinell hardness numbers of most of the test bars. Practically all of the values were well above the required minimum value of 50.

### *Electrical Resistivity:*

The determination of electrical resistivity was made on the specimens used subsequently for tension tests in order to facilitate correlation between these different properties. These determinations were carried out in accordance with the procedure recommended by this Society.[4] Additions of sulfur (Fig. 3) appeared to have little effect on the resistivity of the test bars poured at or below 2100 F.

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[1] Appendix to Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (A.S.T.M. Designation: B 30 – 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 677 (1932); also 1932 Book of A.S.T.M. Tentative Standards, p. 237.

[2] D. Hanson and G. W. Ford, "Investigation of the Effects of Impurities on Copper. Part II. The Effect of Iron on Copper," *Journal*, Inst. Metals, Vol. 32, p. 335 (1924).

[3] H. C. Dews, "The Metallurgy of Bronze," Sir Isaac Pitman and Sons, London (1930).

[4] Standard Method of Test for Resistivity of Metallic Materials for Resistors (A.S.T.M. Designation: B 63 – 29), 1930 Book of A.S.T.M. Standards, Part I, p. 869.

(1150 C.). The bars poured at temperatures of 2200 and 2300 F. (1205 and 1260 C.) had higher resistivities. This increase in resistivity is apparently caused by the high pouring temperature, and not by the sulfur content. The addition of only 0.1 per cent iron greatly increased the resistivity of the alloy in all types of bars regardless of the pouring temperatures.

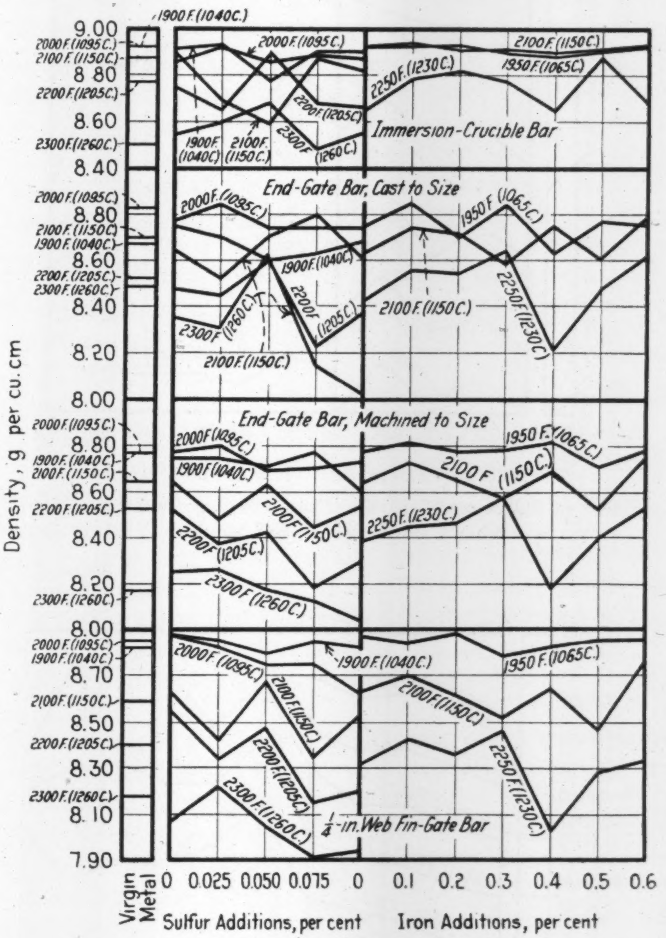

FIG. 4.—Effect of Sulfur and Iron upon the Density.

*Density:*

The density was determined on the ends of the broken tension test specimens by the conventional method of displacement of water. The data, which are summarized in Fig. 4, show that the pouring

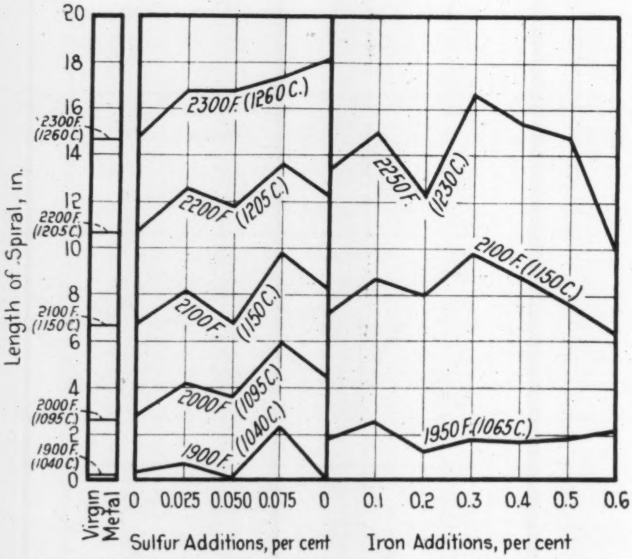

FIG. 5.—Effect of Sulfur and Iron upon the Running Qualities of Red Brass When Cast in Green-Sand Molds.

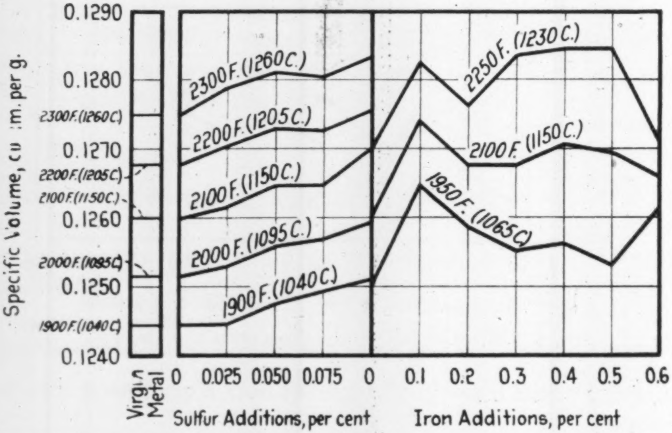

FIG. 6.—Effect of Sulfur and Iron upon the Specific Volume of Red Brass.

temperature is a more important factor than variations in either sulfur or iron content. The addition of as much as 0.6 per cent iron had little influence on the density of the alloys.

### *Running Qualities:*

The running qualities of the alloy containing sulfur or iron were studied by the method described by Saeger and Krynitsky[1] which consists essentially in casting in a sand mold a strip of small cross-section in the form of a spiral. The data which are given in Fig. 5 show that sulfur increases somewhat the running quality of the alloy at all pouring temperatures, and especially at the higher temperatures.

Iron additions to the alloy did not greatly change the running qualities of the alloys. Alloys poured at high temperatures (2100 and 2250 F., 1150 and 1230 C.) showed slightly increased running qualities with an increase in iron content up to 0.3 per cent, but above this, the running qualities decreased.

### *Shrinkage:*

The shrinkage of the alloy was determined by the method described by Saeger and Ash[2] which consists essentially in a determination of the shrinkage of the liquid metal, of the shrinkage during solidification and of the shrinkage in the solid state. The ingot obtained by the immersion-crucible method furnished the data for calculating the specific volume of the liquid alloy. These data, presented in Fig. 6, show that there was a uniform increase in the specific volume with increasing sulfur content for all pouring temperatures. The effect of iron on the specific volume was not so definite or uniform as that of sulfur.

The shrinkage of the alloy in the solid state was determined on each heat poured and found not to be influenced to any important extent by the additions of either sulfur or iron.

The liquidus and solidus temperatures were determined by thermal analysis on the alloy containing the maximum addition of sulfur (0.10 per cent) and of iron (0.6 per cent). These additions did not appreciably influence either the solidus or the liquidus of this alloy composition.

Although the additions of sulfur or iron produce a slight change in the specific volume of the liquid and solid metal, the slopes of the specific volume-temperature curves are the same as those reported for virgin and remelted metal in 1932.[1] There was hence no significant change in the shrinkage.

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[1] C. M. Saeger, Jr., and A. I. Krynitsky, "A Practical Method for Studying the Running Quality of a Metal Cast in Foundry Molds," *Transactions*, Am. Foundrymen's Assn., Vol. 39, p. 513 (1931).

[2] C. M. Saeger, Jr., and E. J. Ash, "A Method for Determining the Volume Changes Occurring in Metals During Casting," U. S. Bureau of Standards *Journal of Research*, Vol. 8, No. 1, p. 37 (1932); *Transactions*, Am. Foundrymen's Assn., Vol. 38, p. 107 (1930).

## SUMMARY

The pouring temperature was found to have a marked influence on the tensile strength, Brinell hardness, electrical resistivity and density of the sand-cast bars, which was decidedly greater than the influence of the added impurity.

The best results, for the physical properties studied, were obtained on the test bars poured at 2100 F. (1150 C.).

The alloy containing sulfur up to 0.1 per cent or iron up to 0.6 per cent meets satisfactorily the requirements of the present specifications of the Society.

In general, the running qualities of the alloy were improved by small additions of sulfur and iron.

The solidus and liquidus temperatures of the alloy were not affected by the additions of sulfur and iron.

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[1] H. B. Gardner and C. M. Saeger, Jr., "Factors Affecting the Physical Properties of Cast Red Brass (85 Cu, 5 Zn, 5 Sn, 5 Pb)," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 517 (1932).

# DISCUSSION

MR. O. E. HARDER[1] (*presented in written form*).—The findings of the authors add valuable data to the available information on the effect of sulfur and iron and of casting temperature on the properties of an extensively used bearing metal.

Some of the data presented seem to require some comments. For example, in the tension tests as shown in Fig. 1 it appears that unexplained variations have been reported. Using end-gate bars cast at 2200 F. (1205 C.), it will be noted that a tensile strength of over 30,000 lb. per sq. in. was obtained with a sulfur content of 0.025 per cent, but when the sulfur was increased to 0.075 per cent the tensile strength dropped to about 18,000 lb. per sq. in., or decreased about 40 per cent, and that at a sulfur content of 0.1 per cent the tensile strength was up to about 27,000 lb. per sq. in.

Similarly with $\frac{1}{4}$-in. web fin-gate bars cast at 2250 F. (1230 C.) the tensile strength increased from less than 28,000 lb. per sq. in. to about 36,000 lb. per sq. in. upon the addition of 0.3 per cent of iron, but was down to about 19,000 lb. per sq. in. with 0.4 per cent iron. Stated another way, increasing the iron from 0.3 to 0.4 per cent appeared to decrease the tensile strength about 47 per cent. No reason is given for these erratic changes in the tensile properties, nor are the individual results questioned by the authors. It would seem from the general trend of their data that there are no such abrupt changes in the properties of these alloys. Also the available knowledge of these alloys does not indicate that such abrupt changes should be expected. These considerations seem to question the soundness of some of the castings, and it would appear that the higher values are more reliable. The question naturally arises regarding the tendency of this alloy to give erratic results in casting practice. I do not have independent data for this particular alloy, but I do have data from a somewhat similar alloy (83 per cent copper, 7 per cent tin, 7 per cent zinc and 3 per cent lead) reported in a recent paper by Carter S. Cole and the writer.[2] In those tests using fin-gate bars cast by two different foundries at temperatures of 1950 to 2160 F. (1065 to 1180 C.) the average tensile strength was 35,100 lb. per sq. in. with the maximum and minimum values for six specimens (three from each foundry)

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[1] Assistant Director, Battelle Memorial Inst., Columbus, Ohio.

[2] O. E. Harder and C. S. Cole, "A Study of Six Bearing Bronzes," *Preprint No. 33-13*, Am. Foundrymen's Assn. (1933).

35,800 and 31,000 lb. per sq. in., respectively. These data seem to indicate that with an alloy close in composition to that used by Gardner and Saeger consistent results can be obtained in commercial foundries. One may, therefore, wonder if in this particular research the foundry practice was largely responsible for the large variations in some of the results obtained.

While the authors determined the density of their alloys as cast, additional interesting data might have been obtained by making density determinations on the alloys after they had been subjected to a compression test, such as compressing at 100,000 lb. per sq. in., which tends to close up the pores and increase the density, as pointed out in the paper by Harder and Cole referred to above.

Finally, a microscopic examination might add valuable information regarding the soundness of individual specimens and might have justified omitting certain data.

MR. H. B. GARDNER[1] (*author's closure*).—The authors appreciate the comments and suggestions of Mr. Harder. The points mentioned were not overlooked by the authors in their study of the data and an endeavor was made to find a cause for the irregularities. No full and satisfactory explanation could be found. As the foundry practice was uniform for all of the heats poured in this investigation, however, it was concluded that the pouring temperature itself was probably the major factor and the results obtained were presented as found. The marked irregularities are found in the curves summarizing the results of the bars made at the higher pouring temperatures, that is, 2200 and 2300 F. (1205 and 1260 C.) for the alloy containing sulfur and 2250 F. (1230 C.) for the iron-bearing alloy.

It is gratifying to have Mr. Harder's work, as shown in his paper, confirm our investigation. In his paper, the test bars reported were poured at 1950 to 2160 F. (1065 to 1180 C.) and his results are in rather close agreement with our data on test bars poured below 2200 F. (1205 C.). This again emphasizes the fact that, while consistent results may be obtained at the lower pouring temperatures, at the higher temperatures the impurities present have less influence on the properties of the alloy than has the pouring temperature itself.

In the paper presented by the authors in 1932,[2] macrographs of the different types of test bars used in this investigation were included. They were made from virgin metal and poured at 2200 and 2300 F. (1205 and 1260 C.). The marked non-uniformity of the fin-gate sand-cast bar was discussed and the suggestion made that a test bar with a more uniform cross-section was more desirable. The micrographs of the same test bars showed strain lines in all of the sand-cast test bars which were most pronounced in the bars poured at the higher temperature. This was considered indicative of straining of the bar by shrinkage and to this was attributed at least, in part, the lower physical properties at the higher pouring temperatures.

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[1] Research Associate of the Non-Ferrous Ingot Metal Institute at the U. S. Bureau of Standards, Washington, D. C.

[2] H. B. Gardner and C. M. Saeger, Jr., "Factors Affecting the Physical Properties of Cast Red Brass (85 Cu, 5 Zn, 5 Sn, 5 Pb)," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 517 (1932).

This further evidence supports the view that the irregularities in the present results should be associated with the higher pouring temperatures rather than simply with the impurities.

# STATUS OF SPECIFICATIONS FOR HYDRAULIC CEMENTS IN THE UNITED STATES[1]

BY P. H. BATES[2]

## SYNOPSIS

This paper is a discussion of the many uses of hydraulic cements and the many service conditions which they must meet. From a consideration of these the question is raised as to the possibility of any one type of cement being able to meet adequately the various demands. The properties of the various types are then presented with a discussion of their adaptability to various services, using as a particular example the conditions which had to be met in the construction of Boulder Canyon Dam and the nature of the cement specified therefor.

The outstanding requirements of the present portland cement standards of the Society are presented and some discussion of their significance is made. There is also given a brief historical summary of all of the revisions of the Society's standards for this commodity, with some explanation of why some of the revisions were made.

Attention is also directed to the tendency of a part of the public to propose standards deviating from those of the Society. Several reasons are advanced for this state and it is suggested that Committee C-1 on Cement recognize the need of several kinds of cement for the various uses and also the inadequacy of some of the specified test methods.

As a result of a series of uncontrollable circumstances, hydraulic cement in the United States is made to meet the requirements of one standard almost exclusively. It is true that this Society has specifications for natural cement and tentative ones for masonry and high-early-strength portland cements. But natural cement is produced in almost negligible amounts; masonry cement is used practically exclusively in mortars for laying brick, tile, stone, etc. Hence, the high-early-strength cements are the only ones used to any degree in competition with standard portland cement. Natural or Roman cements were first produced in this country, and portland cements at first had difficulty in replacing them. After vain efforts to keep the natural cements on the market through "improvements" (largely using various amounts of portland cement as admixtures), the natural cements practically disappeared from the domestic market.

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[1] Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

[2] Chief, Clay and Silicate Products Division, U. S. Bureau of Standards, Washington, D. C.

Today portland cement is employed in a variety of uses and conditions of service that were hardly thought of when it first appeared on the market. In 1900, concrete highways and reinforced-concrete buildings were curiosities. More concrete was used in large masses such as fair-sized dams, and even at that period there was considerable discussion regarding the heat evolved in such structures by the hydration of the cement. Lime mortars were still almost exclusively used for setting masonry, and such materials as plant-made products—block, tile, pipe, etc.—were but little advanced. Further, in the United States, the extended use was small, and the history of service was very brief, due to the lack of the use of concrete for any great period. But now cement has found its way into many unexpected places and has been under observation for such periods that excellent estimates of true success are available. Today, also, many engineers have learned enough about cement, especially of certain physical characteristics, to know that any one type of hydraulic cement will not fulfill all the demands which the great variety of service conditions present.

Let us consider some of the outstanding conditions which concrete must meet in its many modern applications. Possibly the first adverse condition noted was that of aggressive salt solutions, especially sulfates in sea water, mine water, and the white alkali of our West. Several answers to this problem have been offered so far as the cement itself is concerned—cements of high iron content as contrasted to high alumina content, the so-called fused cements or lime aluminate cements as contrasted to the lime silicate cements, portland cements admixed with active siliceous materials, such as the many varieties of natural pozzuolana and blast-furnace slag, and lastly the proper and careful use of the usual portland cement of commerce. But none of these answers is acceptable to more than a few. There are very active proponents of each and just as active opponents. This was well illustrated at the 1931 meeting of the International Navigation Congress in Venice. The reporter on the problem of the use of cement in sea-water construction most positively stated that portland-cement concrete so used disintegrated rapidly and completely. A heated discussion arose and quieted down after many had expressed themselves largely through insisting that their own opinions were better than anyone else's. No one was convinced of anything and the situation was left as muddled as ever.

Retaining walls, conduits, tunnel linings, etc., present a problem through the solvent action of percolating water of even a potable purity. High-limed cements such as portland before or after setting are decidedly soluble in water. If it were not so, setting would not result. The result of the action of water is the rapid saturation of the latter with the production of less basic compounds which are less readily acted upon by the water. If water cannot percolate due to the denseness of the concrete or other means, the continued solution will be prevented. The necessity of preventing percolating waters has been generally recognized, but the waterproofing of concrete or the using of less basic cements is a topic which always arouses discussion, and which in turn seems to change the preconceived ideas of but a few.

Cycles of heat and cold, wetting and drying, freezing and thawing, constitute what may be called weather conditions. Set portland cement has a fairly high coefficient of thermal dilation—how much it varies with different types of cement is not known with any assurance. The coefficient of dilation due to wetting and drying is even greater than that of thermal dilation, but how much it varies with different types of cement is also not satisfactorily known. Attention should be directed also to the fact that much of these same data for aggregates are not available. It is possible that aggregates of large coefficients of dilation are being used with cement of low coefficients and *vice versa*. This difference doubtlessly would be more unfortunate than if both had high coefficients. When the effects of freezing and thawing are more marked than those due to cycles of wetting and drying (at higher temperatures), they are due to the expansion of contained water during freezing. It is possibly not fully realized how much "water voids" are present in good concrete. Take a 6 bag per cubic yard concrete with a 5 gal. per bag water content and assume these are actually in the cubic yard of concrete. If the cement has combined with 20 per cent of its weight of water (which is indeed a very generous estimate and 15 per cent would be more nearly correct), 13.6 gal. of the 30 gal. used would be removed, leaving 16.4 gal. of water free in the concrete. This latter would be equivalent to approximately 3800 cu. in., which is about one-twelfth of a cubic yard. In freezing, water expands about 10 per cent. Can the above concrete withstand having one-twelfth of its volume expand 10 per cent very often? Bear in mind this volume is only that originally occupied by part of the mixing water and is augmented by "air voids" and those resulting from the impossibility of filling all interstices in the coarse aggregate with mortar, and in the fine aggregate with paste. To resist freezing and thawing, the ideal cement would be one that would combine with all the mixing water so that the resulting volume of such reaction would be at least equal to the original volume of water, plus volume of cement, and would develop enough strength to resist the expansive force of the freezing water which had later percolated into the other-than-water voids in the mass.

Having before us some of the outstanding uses to which cement may be placed and the outstanding conditions which it may have to meet, does it seem probable that any one type can always meet the demands? To be most effective in highway construction, it is not necessary that the heat of hydration be particularly low or that marked plasticity be present, or that the cement should be especially resistant to aggressive waters. For such use, good strength is desirable and the earlier this is obtained the better, but the cement should also be resistant to changes produced by both heat and moisture. In reinforced-concrete building construction, the earlier and the higher the strength the better. But since in most cases such structures are entirely protected from the weather, the effect of almost all of the other unfavorable conditions can be disregarded. The setting of masonry demands above all else a cement which will produce a very plastic mortar, but the mortar must have volume constancy both before and after hardening. Mass concrete demands a cement of low heat of hardening, seldom any very marked high strength and generally a good resistance to atmospheric changes, and in some special cases an excellent resistance to aggressive salt solutions. Other demands on the properties of cement by the conditions of its many uses could be amplified considerably, but the above will suffice to illustrate the situation.

Having considered the conditions under which hydraulic cements are used, it would be proper next to give some thought to the type of hydraulic cements available, not only those actually on the market in the United States, but those which world use shows could be made available in domestic markets. Historically, the first to demand attention would be the pozzuolanic cements—those natural and artificial materials which in a fine state of subdivision and sometimes with the addition of accelerating agents have the property of acquiring strength. In certain cases these raw materials require some slight burning to bring out the cementitious properties. In other cases the burning must be quite severe and the class of natural cements and hydraulic limes is reached. Then comes the portland cement group produced by fine grinding of clinkered pre-ground materials. Lastly, there is the group wherein the combination of oxides in the raw materials is such that fusion takes place at a relatively lower temperature than that required for making portland cement. This group includes the fused or high-alumina cements. There are available also various hybrid groups—mixtures of any two or more of those cited.

Do these groups have distinctly different properties which would indicate their meeting certain requirements better than others to a greater or less degree? Unfortunately, in many cases this question cannot be answered with positive assurance. There is no question but that the fused cements generate the greatest early strength and evolution of heat. They are accredited with unusual resistance to aggressive waters. The pozzuolanic cements have the outstanding properties of low early strength, but very steady gain in strength, and an excellent resistance to aggressive waters after they have acquired a certain part of their ultimate strength. The hydraulic limes and natural cements are most satisfactory for their high degree of plasticity, accompanied by low early but excellent late strength. The portland cements are usually held to be the nearest approach to an "all-purpose cement." But the properties of portland cements may vary to a marked degree in spite of their being made to a single standard. Thus, the high-early-strength portland cements are solely the results of some manufacturers noting that by certain changes in plant procedure their usual raw materials could be made to give a product developing much higher early strength than that obtained by their regular output. About 25 years ago, much attention was aroused through the appearance on the market of cements of high iron oxide and low alumina content. These were claimed to be highly immune to the attack of aggressive salt solutions. In this country they have disappeared from the market, due largely to plant manufacturing troubles, and their erratic setting and slow hardening. The demand for plastic or masonry cements has led some manufacturers also to study the improvement of their product with this in mind, through finer grinding, adding plasticizers, etc. Now the demand for a cement of low heat of hardening has led to further study by producers of standard portland cement. It therefore seems that the inadequacy of this one standard cement as an "all-purpose cement" has been definitely recognized for an extended period of years and that the producing industry itself has been decidedly active in trying to determine just how various kinds of portland cement could be produced for meeting pre-determined conditions.

There is one outstanding case before us at the present time—cement for Boulder Dam—and one of less prominence, cement for the highways of New York State. There seems to be a misunderstanding of the conditions at Boulder Dam which have led to the need of a cement evolving low heat during hardening. Boulder Dam will not be a monolithic structure. On the other hand, it is a concrete block dam in which blocks of about 40 ft. in horizontal section cast in place in lifts of about 5 ft. will be used. These blocks at certain times in the course of erection will be grouted together. Moreover, the conditions demand that the grouting be done not when the dam is completed, but at certain periods during construction. Hence, it will not be possible to stand by and wait for the blocks to cool from the higher temperatures resulting from the chemical reactions incident to hardening to a desired temperature so that the volume of the blocks will have diminished to a certain volume. It is true that artificial cooling will be resorted to. But this is not done to prevent the evolution of heat and therefore the hardening. It would be rather unfortunate if cooling prevented too much of the hardening before grouting. If such were the case, it is possible that after the grouting had taken place the hardening and evolution of the heat would be resumed, with consequent possibly too great expansion. The cement for Boulder Dam is designed to have a low final heat of hardening for the purpose of controlling the ultimate volume of the concrete. This should be remembered, and the thought that the special cement is being considered because of its resistance to alkali attack, of its less likelihood of excessive shrinkage, of its low coefficient of thermal dilation, of certain unusual strength qualities, of its workability, and as a panacea for all the faults, granted and assumed to which cement may be heir, should be dismissed from the mind. It will very likely be a most excellent cement for the purpose in question. For all other purposes, it will likely be open to as much question as any other type of cement.

Highway engineers have been much concerned regarding the condition of concrete roads in western New York State. For some time it was felt that the unsatisfactory results were due to some undesirable property in the fine aggregate. The Highway Department of the State, after a critical study, seems to favor the thought that the difficulties may be traced to the composition of the cement which has been changed gradually in the past decade or two, due to the demands for high-early-strength cement. Specifications for cements to meet the apparant exacting demands of western New York should not be based on those prepared to meet other exacting demands. The low tricalcium silicate content of Boulder Dam cements is suggested in view of the high heat of hardening of this compound—not because it has been shown to have any outstanding lack of durability under adverse conditions. The tricalcium aluminate evolves during its hardening much more heat than the tricalcium silicate, and is even more undesirable when the temperature of hardening is to be maintained as low as possible. It is also true that this aluminate is generally considered to be readily attacked by sulfates in solution. But there are no outstanding amounts of sulfates in that section of New York. It would seem that if these two constituents of cement are to be restricted in order to produce a cement for the purpose, it should be on the basis of unsatisfactory volume changes, due either to thermal and moisture variations or to large volume changes resulting from the reaction of the cement and water. It must be confessed that in these respects our knowledge of cements is markedly lacking, notwithstanding the need for these data.

TABLE I.—DIGEST OF A.S.T.M. PORTLAND CEMENT SPECIFICATIONS.

| | 1904 | 1908 | 1909 | 1917 | 1921 | 1926 | 1930 |
|---|---|---|---|---|---|---|---|
| Loss on ignition | Not specified | <4.0 | <4.0 | <4.25 | <4.25 | <4.25 | <4.25 |
| Insoluble matter | Not specified | Not specified | Not specified | <1.00 | <1.00 | <1.00 | <1.00 |
| $SO_3$ | <1.75 | <1.75 | <1.75 | <2.10 | <2.10 | <2.10 | <2.10 |
| MgO | <4.00 | <4.00 | <4.00 | <5.40 | <5.40 | <5.40 | <5.40 |
| Specific gravity | <3.10 | >3.10 | >3.10 | >3.10 | >3.10 | Not specified | Not specified |
| Residue on No. 100 sieve | >8 | <8 | <8 | Not specified | Not specified | Not specified | Not specified |
| Residue on No. 200 sieve | <25 | <25 | <25 | <23 | <22 | <22 | <22 |
| Soundness pats | 28 day in air<br>28 day in water | 28 day in air<br>28 day in water<br>5 hr. in steam | 28 day in air<br>28 day in water<br>5 hr. in steam | 5 hr. in steam | 5 hr. in steam | 5 hr. in steam | 5 hr. in steam |
| Initial set, Vicat | >30 min. | >30 min. | >30 min. | >45 min. | >45 min. | >45 min. | >45 min. |
| Initial set, Gillmore | Not specified | Not specified | Not specified | >60 min. | >60 min. | >60 min. | >60 min. |
| Final set, Vicat | <10 hr. | <10 hr. | <10 hr. | <10 hr. | <10 hr. | <10 hr. | <10 hr. |
| Final set, Gillmore | Not specified | Not specified | Not specified | <10 hr. | <10 hr. | <10 hr. | <10 hr. |
| Tensile strength requirements, lb. per sq. in.[a]: | | | | | | | |
| Neat cement, 24 hours | 150 to 200 | 150 to 200 | 175 | Not specified | Not specified | Not specified | Not specified |
| Neat cement, 7 days | 450 to 550 | 450 to 550 | 500 | Not specified | Not specified | Not specified | Not specified |
| Neat cement, 28 days | 550 to 650 | 550 to 650 | 600 | Not specified | Not specified | Not specified | Not specified |
| 1:3 standard sand, 7 days | 150 to 200 | 150 to 200 | 200 | 200 | 200 | 225 | 275 |
| 1:3 standard sand, 28 days | 200 to 300 | 200 to 300 | 275 | 300 | 300 | 325 | 350 |

[a] Minimum requirements except previous to 1908; in that period the purchaser was requested to indicate the minimum he would accept within the values presented in the specification.

Definition of the Term Portland Cement:

*Previous to 1917.*—"This term is applied to the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3 per cent has been made subsequent to calcination."

*After 1917.*—"Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion of an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination excepting water and calcined or uncalcined gypsum."

NOTE.—Between 1909 and 1917 the Test Methods included a description of the making of 2 in. standard sand 1:3 mortar cubes but no values for the strength of these were given.

The American Society for Testing Materials' activities in the field of cements originated in 1900. In 1904 it adopted its first set of standards. In Table I are briefed certain of the requirements adopted then and their history through the several revisions to date.

A consideration of the individual requirements of the Society's specifications from the viewpoint of their significance would possibly be of some interest. Throughout the thirty-two years of the Society's activities there has been one change in the definition of portland cement. Do many realize the full significance of the present definition? Do they realize that portland cement is a clinker ground with two or three admixtures—water and crude gypsum or water and calcined gypsum? Further, is the full significance of the clause "excepting water and calcined or uncalcined gypsum" realized? What is calcined or uncalcined gypsum? To answer this question, access is had to the standards of this Society. A.S.T.M. Standard C 22 - 25[1] states that, "no material may be considered gypsum within the meaning of this specification which contains less than 64.5 per cent by weight of $CaSO_4 \cdot 2H_2O$." Nothing is stated as to what the remaining 35.5 per cent may be. Therefore, a manufacturer may add to each 93½ lb. of clinker 6½ lb. of a "gypsum" containing up to 2.2 per cent of calcium chloride, diatomaceous silica, pumicite, tufa, soluble silicates, ground limestone, tannic acid, and any "what not," and still be furnishing a cement meeting the definition of this Society. Are those manufacturers who add sand or iron oxide to their raw mixture making portland cement—according to the definition? They are not, since argillaceous and calcareous raw materials alone are permissible and neither sand nor iron oxide can be classed as such.

How many recall the definition in use before January, 1917? The last portion of that definition read, "and to which no addition greater than 3 per cent has been made subsequent to calcination." Before that time when the user was already "admixture conscious" it was entirely proper to add in the grinding of clinker 3 per cent of almost anything whatsoever. The specification did not state what the 3-per-cent addition might be. There is much talk now of the good old kind of cement. Possibly some of this good old cement had as an addition certain of those materials which now so irritate manufacturers when they are suggested instead of "calcined or uncalcined gypsum." Historically, portland cement need not contain calcium sulfate to be classed as such.

Loss on ignition and insoluble residue are supposed to indicate adequacy of burning, too long storage or adulteration. Would it not indeed be a "sad" cement that would be so inadequately burned as to exhibit even half the limits of these requirements? What manufacturer would hold a cement in storage so long as to attain even half the limits of the ignition loss? Bagged cement in warehouses may attain the limit of ignition loss, but how frequently is such cement tested? Ground raw materials would surely first suggest themselves to a manufacturer who might desire to adulterate his product and the limits in the specification would restrict them to a somewhat modest amount—if the use of any amount of adulterant can be considered modest. However, there are a number of common materials, as blast-furnace slag, which might be used and to which these requirements would not apply. Is there sufficient justification to maintain these two requirements, particularly when the cement must meet a strength requirement?

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[1] **Standard Specifications for Gypsum** (C 22 - 25), 1930 Book of A.S.T.M. Standards, Part II, p. 94.

There seem to be sufficient data available to indicate that there should be limits on both the $SO_3$ and MgO. Since any harmful effect of these would only be indicated after the lapse of rather long periods of time, reliance cannot be placed upon the strength at the ages at which cement is tested by the standard. The question as to what limits should be placed upon these two constituents has been a matter of discussion and study from the earliest history of cement. It seems to be as far from conclusive settlement as ever.

At the present time any argument favoring the continued use of the No. 200 sieve in a standard would seem to be very similar to the sieve itself—decidedly full of holes. But much could be written about this requirement. At one time it occupied the attention of the Society's Committee C-1 on Cement to the exclusion of all else and threatened to bring about a difference between the Society's standard and that of the Federal Government. New types of grinding equipment have about removed the question of fineness from the standards picture, if one is satisfied with the indication of the strength requirements, and is not afraid of too fine a cement. In any case, the sieve itself is practically obsolete and attention should be directed to other devices for measuring fineness. With all the discussion, however, regarding fineness, reference to Table I will show that in the history of the committee, the fineness has been changed only from the original 25-per-cent residue to the present 22-per-cent residue.

The next item of concern in our specification is that of consistency. There has been practically no change in this since the first efforts of the Society, notwithstanding the studies which have been made. For the neat pastes, the procedure is apparently adequate, but due to the present very fine grinding, the values to be used in the mortars are most faulty. There seems to be much to commend the suggestion of the use of the same amount of mixing water for all cements.

Our present time-of-set methods and requirements seem satisfactory, if one will neglect the much mooted question of "grab or hesitation" set and the eternal question of what constitutes "appreciable indentation." It seems strange that this "appreciable indentation phrase" should have such a grasp on life, when it could be so readily done away with. "Grab or hesitation" set is ignored and hence apparently does not exist, according to the Society's specification—hence we have the reason for some specifications that do not ignore it and hence do acknowledge its existence. Again, it would be a simple matter to take care of this apparently modern (?) fault in cements in any standard. But doing this would acknowledge that cements may have such a fault, which seems to be a decided "rub" at the present time. Before leaving this topic, may we reminisce enough to recall the old "squabble" (used advisedly) over Vicat *versus* Gillmore needles for determining the time of set. The affair has been somewhat mellowed with age, but the outcome is quite illustrative of how things may happen to be in any specification, and in this case two things—both needles. Since both sides won in this discussion, both were pleased—in at least that the other side did not solely have its way.

More changes have been made in the requirement for tensile strength than any other item of the standard. This has really represented a vain effort to keep pace with the increased strength which concrete tests have indicated cements were developing. It is too bad that those having to do with the specifications have not long since definitely abandoned the briquet and the thought that there is a fixed relation between tensile and compressive strength at all ages. All data to date show that concrete in tension (whether the tensile stresses are induced by a direct pull or by bending) gains the greater part of its ultimate tensile strength at a much more rapid rate than its compressive strength. Hence, after a month or two, there are relatively slight gains in tensile or transverse strength, whereas the compressive strength is still increasing handsomely. It is gratifying, however, to note the interest being taken in studying the plastic mortar compression cubes. The results seem to indicate that such a test specimen comes nearest to the ideal of testing the cement in a concrete cylinder. One of the Western states now purchases its high-early-strength cement on the basis of the strength of a 6 by 12-in. concrete cylinder, and another state is about to include a similar test for its standard cement.

There is much discussion now regarding the advisability of including in specifications, requirements limiting the amount of certain compounds calculated from the chemical analyses. There is no doubt but that the results of such calculations do furnish a most interesting additional picture of the nature of cement. Hence, there is much to commend such calculation. But certain considerations must be remembered. Above all, the inherent errors in chemical analyses must not be lost sight of; furthermore bear in mind such errors are multiplied in some cases as much as ten fold in carrying out the calculation. The inadequacy of analytical methods must also be considered—thus cement will absorb water with the likelihood that the major portion of the water will form hydrated lime. This latter in the analytical procedure used will appear as free lime, with consequent apparent reduction in the amount of tricalcium silicate actually present in the original cement. It is not known how to figure the combinations of the oxides of sodium, potassium, magnesium, titanium, phosphorus, etc., and carbon dioxide. Hence, they are neglected, although they are present invariably to the extent of one per cent, and frequently as much as two per cent or more, combined with lime, alumina or silica, and therefore should very materially alter the values obtained through calculations. Caution, therefore, should be used in interpreting the results of these calculations, and no fine distinctions should be drawn between cements showing a couple of per cents difference in the amounts of the several compounds.

Attention should also be directed to the fact that we are still largely ignorant of the properties of the four major compounds (tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium alumina-ferrate) derived through the suggested calculations. In relatively very few cases the heat developed by either of these through hardening has been calculated from the heats observed during the setting of cements and from the calculated amounts of compounds in the cements. These data, however, must be confirmed with those obtained directly from the hardening of the pure compounds. We know relatively how much each contributes to the strength at various ages. But here confirmed data regarding all the other properties largely stop and leave us in a rather precarious position to make any positive assumption regarding the positive or negative values of the compounds for the various conditions of usage. There is a marked insufficiency of data regarding the part played by each in the broad question of volume changes induced either by thermal or chemical effects. (In this paper not as much emphasis has been placed upon volume change as there should be. This is due to the reluctance to discuss a matter which has not received the attention in research or testing that it must have in the future.) There is no doubt but that in the not too distant future there will be a quantitative volume change requirement in our specifications, replacing our present inadequate soundness test.

Before leaving the subject of the constituents of cement, it would be well to call attention to a tendency to enhance the value of the iron oxide when used to replace alumina and at the same time depreciate the value of alumina in cements. But the best information available would indicate that the result of the reaction of the water with the tetracalcium alumina-ferrate is identical to that of water and the tricalcium aluminate—plus the additional formation of hydrated iron oxide and hydrated lime, the formation of which in portland-cement concrete gives so much concern to so many.

From this discussion of the needs of cement in order that it may render satisfactory (if not permanent) service and the significance of the test requirements, it is rather evident that much is lacking in the latter, even though we acknowledge the difference that often very properly exists between acceptance tests and tests indicative of service. The growing tendency on the part of users to prepare specifications, covering rather restricted uses, differing from those of the Society in spite of the revisions by the Society, indicates conclusively that the consuming public is not satisfied with matters as they have been progressing. It seems very necessary for Committee C-1 to give more cognizance to the existing conditions, to recognize the need of several cements for the many different uses to which cement is put, and to acknowledge the inadequacy of some of our test methods. Specifically, it must recognize the difference between the conditions prevailing in constructing a dam, a road, or a reinforced building, etc., and in the conditions in the after-life of these. Since these conditions are so different, it must accept the fact that we will need cements that are able to harden with minimum heats of hydration, that do not have large coefficients of thermal or moisture dilation, that are resistant to attack of aggressive waters, that do resist freezing and thawing, that for some uses have very high strength, and that for other uses have marked plasticity. Since it seems impossible to have more than one or two of these properties developed to any major degree in any one cement, it follows that there should be prepared recognized specifications for at least six cements, namely, high-early-strength cements, plastic cements, those of low heat of hardening, those having low volume changes, those offering resistance to moisture and aggressive solutions, and at least two of somewhat the nature of our present standard portland cements, one of these two being lower in lime and higher in silica than the other.

The test methods which need most attention are more detailed chemical requirements, either no measure of fineness or one that indicates decidedly more than does the sieving test, a measure of thermal and hardening dilation, and a thorough upheaval in the strength requirements, entirely eliminating the use of the briquet of standard sand and standard consistency, and substituting therefor, if we are not bold enough to use a concrete test, a cube for compression testing made of graded sand and of plastic consistency.

Here this discussion would have ended had there not been received about the time it was being completed a paper by Mr. A. Lundteigen, whose connection with the cement industry dates from the time when cement was made by burning briquetted raw materials in upright kilns. This paper appears in the transactions of one of our leading engineering societies. It occupies but eight pages, but is followed by twenty-seven pages of very earnest discussion on the part of many. It seems worth while to present a few extracts from Mr. Lundteigen's paper and a few from the discussion presented by Mr. Robert W. Lesley, the senior member of Committee C-1 and a charter member of this Society. The first sentence of the paper is, "A number of years ago the Society of German Portland Cement Manufacturers published an article claiming that good portland cement could not be improved by admixtures." The following are also abstracted from the paper:

> "Whenever failures in concrete works were reported, they were attributed either to poor workmanship in the construction or to bad cement . . ." "A cement was regarded bad when it contained, through careless manufacture, too much free lime, magnesia, or sulfuric acid." "A closer investigation of the effect of sulfates in concrete was made . . . The results are, in short, that the amounts of alumina play an important part . . ." "It is different, however, where the concrete is to be used under water, be it fresh or salt. Then high-limed high-grade cements will not only prove inferior but dangerous." "The harmful effect of free lime has always been well enough known, but there is at the present day, with improved machinery and methods of manufacture, no excuse for such free lime . . ."

The following are abstracted from Mr. Lesley's remarks:

> "The obvious result of the tendency toward specifications requiring on the one hand very great fineness and requiring on the other hand very great tensile strength . . . has been to lead producers of cement toward the danger line, so far as the ultimate safety of the product is concerned. In order to accomplish the desired result indicated by the divergent elements of these latter

specifications, the proportion of lime in the cement was increased 3 or 4 per cent . . ." "Meanwhile, specifications were still raised . . . and extraordinary fine grinding still further increased. This entailed still higher lime and the discovery was made that this overlimed portland cement would sometimes crack at short periods, and sometimes go to pieces at long periods." "Gypsum or sulfate of lime in its crude or calcined condition is about as cheap as most of the materials which, in the course of manufacture, ought to enter into cement . . ." "The German Society of Portland Cement Manufacturers cites in support of the fact that portland cement does endure in salt water, works laid down as far back as 1840, where the character of the cement was very different from the character of the portland cement made to meet the abnormally high specifications of the present day."

Now the climax—Mr. Lundteigen's paper was presented in the *Transactions* of the American Society of Civil Engineers, April 7, 1897. Statements similar to every one of these quotations have been repeatedly presented in meetings of Committee C-1 and like organizations within the past few years. Such citations should serve as bearings and indicate conclusively that instead of advancing on tangents we are in many respects progressing around circles and are but a degree or two beyond the position found when bearings were last taken.

# DISCUSSION

Mr. G. O. Gardner[1] (*presented in written form*).—In this paper Mr. Bates has clearly given an outline of the status of the cement specifications. While I think as a whole it is proper for not only the producers but the consumers to proceed cautiously with changes in the specifications, we have a growing conviction among the consumers of portland cement that the present standards of testing are inadequate and of little value so far as determining the probable concrete strengths and other qualities that the cement shall produce.

There are a few points in this paper which we wish to discuss. Mr. Bates states: "There is much to commend the suggestion of the use of the same amount of mixing water for all cements." We can agree with him to the extent that if this were adopted, a given cement would be tested the same in all laboratories, which is not the case under the present methods of testing. However, different cements require different quantities of water to produce the same consistency of mortar or concrete in the same mixture of cement and aggregate. This would result in the testing of different cements under different conditions and not obtain the relative qualities of the cement under test for a given condition of use.

Experience of mortar tests indicates that consistency of mortar has a large effect on the strength though the water-cement ratio is the same for the different consistencies. This is also true in concrete. Furthermore, the cement requiring the least water to produce the desired consistency would probably give the most desirable concrete with respect to durability, volume changes, permeability, etc., because of the lower water-cement ratio.

A testing procedure based on the same quantity of water for all cements, without regard to consistency, might promote the production of cement requiring more water to a given consistency in a given mix, since experience shows that with a given water-cement ratio the stiffer mortar within certain limits gives higher strength results, and it would seem that we should not go to a fixed water-cement ratio until we have proved beyond doubt that it is impossible to measure consistency, and have a standard method for such a determination.

I quite agree that "it is too bad that those having to do with specifications have not long since definitely abandoned the briquet," as there certainly is much data which bears out the fact that the tensile strength as measured by the briquet is not a measure of concrete strengths.

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[1] Superintendent, Ash Grove Lime and Portland Cement Co., Chanute, Kans.

Probably one of the chief reasons why a consistency test is not easily standardized is because of the fact that many cements do have what Mr. Bates has called a "grab" or "hesitation" set. In all of the cooperative tests in which we have participated, some of the cements in each instance have had this characteristic. I do not think that this quality is detrimental at all so far as the cement is concerned, but only that it does interfere with the testing of cement. It does seem to me very probable that this particular quality of cement can be eliminated.

MR. IRA PAUL[1] (*presented in written form*).—Mr. Bates' paper is a timely discussion on the subject of cement specifications. I believe that engineers and chemists are beginning to recognize that cement for whatever purpose used must have other characteristics and properties in addition to strength. From the chemical viewpoint cement is considered an impure chemical or combination of chemical compounds, yet our present A.S.T.M. specifications do not make any reference to the important constituents present in that product which will produce strength and durability. A considerable amount of research work has been devoted to the physical angle of the problem. Attention is now being focused on the chemical aspects and its relationship to the physical characteristics. Mr. Bates, who was instrumental in drawing up the specifications for the cement used in the Boulder Dam, endeavored to incorporate the important physical and chemical characteristics in these specifications, and I see no reason why that type of cement cannot be used with success in concrete pavements. As a matter of fact, recent tests conducted by me with this type of cement have proved that we can obtain concrete equal in strength at the same ages as that produced with high tricalcium silicate cements. At the same time it is my firm belief, from the data assembled in a recent survey I made on concrete pavements in New York State, that the composition of the cements has been a contributing factor on the durability of concrete pavements. Action is now pending in our department for a modified cement specification which includes limiting the percentage of the tricalcium silicate and tricalcium aluminate constituents.

We have used both the low and high tricalcium silicate cements, containing varying percentages of the tricalcium aluminate compound, for almost two decades, and have found that the high tricalcium silicate cements in combination with such sands as are highly absorptive, and generally break down in the magnesium sulfate accelerated soundness test, but fail to show this property with the sodium sulfate test, have failed to give satisfactory service in concrete pavements. Whether this is caused by unsatisfactory volume changes, as Mr. Bates seems to think, or any other reason, these are the facts and we must face them in spite of the lack of sufficient laboratory data. If laboratory tests fail to check our field service information, there must be something radically wrong with our present laboratory methods of tests. This is also true of our standard tensile mortar briquet test, because that does not measure the quality of the cement. I have found no relationship between the tension test on cement and the strength tests on concrete.

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[1] Associate Laboratory Engineer, New York State Department of Public Works, Division of Engineering, Albany, N. Y.

As regards the chemical analysis of cement and calculation in terms of potential constitution, I believe Mr. Bates' objection can be overcome: first, by standardizing the method of chemical analysis and the preparation of the sample, and second, by having the tests run by a competent, experienced chemist. I see no objection to a specification on the basis of oxide composition provided it is expressed in the form of a "lime silica index." Such a method was brought to the attention of the Society's Committee C-1 on Cement by Mr. G. A. Saeger in 1928.[1]

MR. THADDEUS MERRIMAN[2] (*presented in written form*).—Among the more significant of the considerations to which Mr. Bates refers is that of the recent tendency to consider that a cement contains certain compounds simply because it is possible to compute them from a chemical analysis. Such computations come to be looked upon as truth, but the best they show is that a particular cement *may* contain the compounds the computation indicates. The writer is, therefore, in complete accord with the author that: "Caution, therefore, should be used in interpreting the results of these calculations, and no fine distinctions should be drawn between cements showing a couple of per cents difference in the amounts of the several compounds." The compounds to which the analyses are computed are known only in the laboratory where they are made under controlled conditions, with refined apparatus and from pure materials. But the rotary kiln works on a heterogeneous mixture which contains many impurities; the temperature fluctuates within comparatively wide limits; the time during which the mix is in the kiln depends on how rapidly it is rotated; the character, composition and fineness of the raw mix is not always constant; the cooling and quenching conditions vary as also does the fuel with which the kiln is fired. Under such conditions, it is not possible to say that because the pure compounds can be made in the laboratory and because the microscope discloses some of them in the clinker, therefore, the cement contains these compounds and only these. To draw such a conclusion is to endow the rotary kiln with an efficiency far superior to that of the laboratory where the compounds in question can be made only by the exercise of the highest care and skill.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 238 (1928).

[2] Chief Engineer, Board of Water Supply, New York City.

The question to which we need an answer is: "What percentage of the computed compounds are actually contained in a particular cement?" The limitations of the method and the assumptions which led to the conclusion often pass into the background. We must keep in mind the limitations of our assumptions.

MR. H. A. AMBLER[1] (*presented in written form*).—Mr. Bates has suggested many changes in the specifications for portland cement, and there is a great deal of merit in his proposal to discard such obsolete requirements as loss on ignition, insoluble residue, and 200-mesh fineness, and to rely on a strength requirement in their stead to show that there has been no under-burning, prolonged storage or adulteration of the cement.

If the proposed changes be made, the strength requirement will be even more important than at present, and the briquet should certainly be abandoned for a test specimen that will more clearly represent the strength quality of the cement. Compressive strength is much more indicative of the concrete-making value of a cement than is the tensile strength, and cements should logically be rated according to their compressive strengths.

That the compressive strength of concrete cannot be predicted from the tensile strength of the cement is unquestioned. A cement that has a low tensile strength may have a high compressive strength, and the reverse is also true. With certain cement compositions, extreme finish fineness is required if the present A.S.T.M. tensile strength specifications are to be met, yet these cements may have abnormally high compressive strengths. Manufacturers of cements of this type are heavily penalized by the present specification. On the other hand, we have in our laboratory tested samples of high-early-strength cement that easily passed the tensile strength requirement of the A.S.T.M. Tentative Specifications for High-Early-Strength Cement, and yet had compressive strengths no better than that of several brands of portland cement tested simultaneously. In cases of this kind, the consumer is penalized by the present specifications.

The concrete cylinder is the ideal compression test specimen, and high-early-strength cement is sold in this district on the basis of the strength of 6 by 12-in. concrete cylinders. We have found this requirement much more satisfactory than a tensile strength requirement, for, while the early tensile strength of different lots of cement varies widely, the corresponding concrete compressive strengths are remarkably uniform. However, there are serious objections to the universal use of such a test specimen. Aggregates from different sources not only vary in their absorption, but, even though of the same grading and apparently sound, may give concrete strengths that vary over a wide range. These variations would necessitate the use of a standard aggregate, which would make testing costs prohibitive.

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[1] General Superintendent, Superior Portland Cement, Inc., Concrete, Wash.

Plastic mortar tests have given very promising results, and if current investigations prove that concrete strengths can be predicted from such tests, then, by all means, let us abandon the briquet test, and adopt a compression test which will show us the true strength value of the cement.

MR. L. W. WALTER.[1]—Mr. Bates has had as good an opportunity as anyone in America to review the results of investigation and research directed in the study of cement and without question is well informed. His paper would indicate that he is open-minded. Others of the Society's Committee C-1, representing both producing and consuming interests, are also open-minded and will, I believe, be willing to sponsor and support appropriate revisions from time to time. So long as we may feel that way about it, I think the atmosphere surrounding the committee is favorable to constructive work and instead of the individual consumer stepping out and writing a new specification after his own ideas, it would be better for him to refer to Committee C-1 his views and his reasons for desired changes. I hope the consuming interests will do that.

MR. J. C. PEARSON.[2]—I do not believe that the majority of the producers would be averse to any reasonable change in the specifications that might be required for specific uses of cement. However, in many instances the special requirements of a user may be met by careful selection of a mill product, rather than by a radical change in the processing. When a cement of different composition or radically different properties is required, it is both inconvenient and expensive to make a change in the plant, when the plant is designed to produce regularly a single product. Such a change can be made economically only when a large quantity of the special material is wanted. But in any case, there is probable waste of material during the change, and always the problem of storage and of further waste in shipping. Therefore, the manufacturer wants assurance that the special requirements are well established and warranted and not merely based on the whims or notions of individual users. I think if that is once understood generally, then the producers would not oppose any such move. Of course, Mr. Bates refers to six different types of cement, of which at the present time we are able to distinguish only two by our accepted methods of test. That is another difficulty and it is quite a serious one.

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[1] Inspecting Engineer, Erie Railroad Co., Jersey City, N. J.

[2] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

MR. R. S. GREENMAN.[1]—This question of a revision of the cement specifications I believe is due to having a wrong idea of just what we expect ultimately of a cement. One of the reasons for some suggestion for modification of the specifications is due to the fact that there are people who fear that the cement is not as durable a product as can be produced. I think we have been laying too much stress on getting a strength result at an earlier period than is perhaps necessary for general uses. I do know that there are occasions where high early strengths are necessary, but there is also a feeling on the part of a great many users that the ultimate is better secured by a cement which will live long but may not show a great activity in its earlier stages. I should like to emphasize that we must get away from giving strength the high place which it has.

After all, we do know that some of the earlier cements gave results which were entirely satisfactory after long periods of time—fifty years or more. If, then, as in the case of the State Highway Department of New York, they have some roads that are going to pieces in short periods, it is perfectly natural for them to feel that there is something wrong somewhere. I know that it has been attributed to one thing and another—aggregates, processes of use, lack of curing and any number of reasons. They have had their experiences there but I am thinking also of the use of the product in general structural work. I have noted, for example, a very decided erosion in some artificial stone on a very fine building so that the aggregates are more and more exposed. While the stone is not disintegrating to the extent that it is going to be a failure and collapse, it is going to show up very soon to the extent that people will say: "Give us natural stone masonry." We should recognize that long life of the product in which the cement is used is more important than the immediate use of that product.

THE CHAIRMAN (*Mr. R. E. Davis*).[2]—We have long been strength conscious; we must now become durability conscious. At the same time we must appreciate that durability under one condition does not necessarily mean durability under another condition. The condition we meet in buildings is quite different from that in highways, and the condition we meet in highways is quite different from that in dams.

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[1] Consulting Concrete Engineer, Albany, N. Y.

[2] Professor of Civil Engineering, In Charge of Engineering Materials Laboratory, College of Engineering, University of California, Berkeley, Calif.

MR. M. H. ULMAN.[1]—Mr. Bates' discussion on mortar cement did not cover one very important detail, which is brought to the attention of testing engineers in many instances by the architect's specifications. I refer to the efflorescent properties of mortar cement. I appreciate that with a high lime content, which occurs in mortar cement, it is difficult to eliminate efflorescence. This efflorescence could probably be considerably reduced by requiring a dense mortar, and I am suggesting in specifications for mortar cements that a note be added requiring a dense mortar or specifying the proportions to be used to overcome this objectionable feature of efflorescence.

Mr. Bates further discussed using the same amount of mixing water for all cements. This is not desirable, for the amount of mixing water should be dependent on the fineness of the cement. I am, therefore, suggesting the continued use of a consistency requirement so as to regulate the amount of mixing water.

Mr. Bates further discussed the undesirability of tensile strength in controlling the quality of cement. I am of the opinion that since the Reference Laboratory has been making inspections and offering suggestions at the various laboratories, all laboratories that have been inspected and are now following the methods as recommended by Committee C-1 can obtain very uniform results when comparing the test data of the various laboratories.

I agree with Mr. Bates' statement that tensile strength does not give information as to the ultimate strength of the concrete. However, I wish to state in view of a considerable amount of testing in which we have cooperated with other laboratories that the tensile strength of cement is an index of the strength of the concrete. We have found particularly at the early ages that high tensile strength cement gives higher concrete strength and that low tensile strength cements will furnish low concrete strengths at the early ages. The tensile strength of cement has furnished valuable information in the past in our studies of this product, and in view of the data which have so far been accumulated it would be very difficult to evaluate or reconcile the old test data in our present or future studies of concrete. This would be particularly true if this test were entirely eliminated and any new ones substituted, which would not furnish data comparable to the former methods of test.

MR. HUBERT WOODS[2] (*by letter*).—All who are interested in cement specifications, or in improvements in cements, should be grateful to

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[1] Assistant Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

[2] Chemical Engineer, Riverside Cement Co., Riverside, Calif.

Mr. Bates for his straight-forward discussion. The conviction is growing, especially among users, that *one* standard cement cannot be (or at any rate, is not) most suitable for all purposes.

We agree with Mr. Bates that Committee C-1 should give more cognizance to the existing conditions; further, that the committee should cooperate with the proper agents of the cement industry to determine what cements are best suited to particular uses, and should then frame tentative specifications therefor. It is pretty obvious that not enough is known about the properties of various cements to permit this to be completed very soon. This, however, merely emphasizes the importance of the research work which the Portland Cement Association and others have done, and are doing.

The cement industry should be vitally interested in studying the questions raised by Mr. Bates. If it does not maintain a position of leadership in studying different cements, and in using the results of such studies as bases for further specifications, both the industry and its customers may suffer as a result of the numerous and uncoordinated specifications, oftentimes based on erroneous or incomplete information, which may arise.

After calling attention to the lack of knowledge of the properties of the four major cement compounds, Mr. Bates goes on to say: "In relatively very few cases the heat developed by either of these through hardening has been calculated from the heats observed during the setting of cements and from the calculated amounts of compounds in the cements. These data, however, must be confirmed with those obtained directly from the hardening of the pure compounds." We feel that these quoted remarks may possibly be misinterpreted to mean that it is necessary, before placing reliance on the heat data derived from cements, to determine the heat evolving properties of the pure compounds, and to assure ourselves that the two sets of data are in agreement. However, it has not been shown that in actual cements, each of the individual compounds acts entirely independently of the presence of the others. Hence a study of the heat evolution of the separate pure cement compounds may result in data not agreeing with those derived from observations on actual cements. This, however, in no wise invalidates the latter data, since these were obtained under totally different conditions. Although there are undoubtedly good reasons for desiring to know the properties of the pure cement compounds separately, nevertheless heat data derived from cements are of more direct significance for use in predicting the heat evolution of actual cements.

# THE EFFECT OF ADDING A SILICEOUS MATERIAL TO PORTLAND CEMENTS UPON VOLUME CHANGES, COMPRESSIVE STRENGTH AND HEAT GENERATION

By R. W. Carlson[1] and G. E. Troxell[2]

## Synopsis

This paper points out the possible advantages to be derived from the blending of siliceous materials with portland cements, particularly with regard to mass-concrete construction. The results of a series of tests to determine volume changes, compressive strength and heat of hydration of blended cements are presented.

The variables in these tests are calcination temperature of the silica, chemical composition of the cement with which the silica is blended, curing condition, method of blending, and proportions of silica and cement. It is found that these variables determine to a considerable extent whether or not the blending of silica with cement leads to favorable or unfavorable properties of mortar.

In general, volume changes of mortar due to loss of moisture are shown to be lower at the early ages and higher at the later ages for blended cement than for plain cement. The tendency of the silica to increase the contraction at later ages appears to be least for the high-lime cement, for preliminary curing under mass-concrete conditions, and for silica calcined to incipient fusion.

The 7-day compressive strength of mortar is shown to be lower for all blends of silica and cement than for plain cement, but the strengths are more nearly equal at later ages. As much as 30 per cent of silica may be blended with a high-lime cement without appreciably reducing the mortar strength at the age of 3 months.

It is shown that the effect of blending silica with cement is to reduce the heat of hydration, but not by the full percentage of cement replacement.

It is concluded that the test results show sufficient promise to justify further investigation, covering a wider range of materials and conditions of blending, together with a study of additional properties such as economy of cement manufacture, and workability and durability of concrete.

## Introduction

As an ideal, the engineer strives to produce a concrete of such characteristics that actual stresses may be computed by considering only the applied loads, including the weight of the material, without reference to the effects of temperature and moisture conditions upon the mass. In practice, however, he would be satisfied if the stresses due to changes in temperature, moisture and degree of hydration were so small that they would not disrupt an extended mass of concrete designed without contraction joints. Whatever progress he can make in this direction tends toward structural stability, economy, and permanence.

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Scientific search for the ideal concrete demands an investigation of the effect of each important variable, such as chemical composition and fineness of cement, water-cement ratio, richness of mix, gradation and properties of aggregate, method of compaction, etc., upon each important property of the concrete such as volume changes, strength, heat of hydration and durability. Much of this work has been accomplished but much remains to be done. One variable which has been investigated extensively with relation to compressive and tensile strengths, but only slightly with relation to other properties, is the blending of siliceous materials with cement.

During the hydration of portland cement lime is liberated; the higher the lime content of the cement the more lime liberated. This uncombined lime is undesirable because it is soluble in water and thus may be leached from the concrete. Finely divided silica is capable of combining chemically with the lime at a rate which depends upon the curing temperature and upon the condition of the silica; thus in the presence of silica the uncombined lime is made to serve in a secondary cementing or solidifying action. This suggests the possibility that the substitution of a part of the cement by a siliceous material may produce a concrete of improved qualities.

Aside from the effect of silica upon the quality of the concrete, there are other possible advantages which may be important. It is fairly well known that there can be economies in manufacturing a cement which is a mixture of portland cement and silica, because of the smaller amount of clinker which must be burned and the relative ease with which the mixture may be ground. New developments in portland cement for mass concrete now suggest a still greater economic advantage for the cement-silica blend. The grinding of a portland cement of low lime content, suitable in itself for mass concrete, is considerably more difficult than the grinding of a cement higher in lime content. As will be explained in detail later, the best properties of blended cements are obtained when silica is blended with a cement of high lime content. Thus the possible advantage of ease of grinding due to working with a reasonably high-lime clinker is added to the economy in burning and the beneficial effect of the silica upon grindability.

It is now recognized that strength alone is not a satisfactory measure of quality of concrete and that such properties as durability, volume changes and heat of hydration are also important. In most structures, compressive strengths far above the structural requirements are obtained. It might therefore seem feasible to reduce the cement content and thus to sacrifice the extra strength in favor of a lower heat of hydration and lower volume changes. No large reduction in cement content can be made, however, because workability, water-tightness and durability must be maintained at safe levels. The problem, then, is to obtain the best combination of properties, with perhaps less emphasis on strength and more emphasis on such properties as contribute toward reducing volume changes and the tendency to disintegrate with time.

## SCOPE OF TESTS

The tests herein described have been conducted in the Engineering Materials Laboratory of the University of California to determine the effect of using plain portland cement in comparison with cement-silica mixtures upon the following important properties:

1. Volume changes of mortar under various curing conditions.
2. Compressive strength of mortar under various curing conditions.
3. Heat of hydration of cement.

Durability tests have also been planned but these remain to be made. The mixtures were made from three cements of varying lime contents (the chemical compositions of the cements thus differing principally in the percentage of tricalcium silicate), using three percentages of silica in four different states due to variations in temperature of calcination. The siliceous materials were introduced as a replacement for an equal weight of cement both by intergrinding with the cement and by merely mixing with the cement. The tests are being made on specimens cured under standard conditions of moisture and temperature, and also on specimens cured under conditions simulating those which exist in mass concrete.

## PREPARATION OF BLENDED CEMENTS

The three portland cements (herein designated as "low-lime," "normal-lime," and "high-lime" cement) which were used both plain and blended with the siliceous materials were chosen from a group of 25 cements manufactured for the cement investigations for Boulder Dam. Each of the selected cements has a low content of tricalcium aluminate. The chief difference between the cements, with respect to the effect of blended siliceous materials, lies in the tricalcium silicate content; the low-lime cement contains 33 per cent, the normal-lime cement contains 55 per cent, and the high-lime cement contains 70 per cent of tricalcium silicate. The compound compositions are shown in the following table:

| | LOW-LIME CEMENT | NORMAL-LIME CEMENT | HIGH-LIME CEMENT |
|---|---|---|---|
| Tricalcium silicate, per cent | 33 | 55 | 70 |
| Dicalcium silicate, per cent | 45 | 23 | 10 |
| Tricalcium aluminate, per cent | 7 | 6 | 0 |
| Tetracalcium aluminoferrite, per cent | 12 | 14 | 17 |

The siliceous material blended with the cements is a volcanic ash from a natural deposit near Fresno, Calif. It occurs finely divided, with 98 per cent passing the No. 200 sieve. Its principal constituents are 75 per cent silica, 15 per cent alumina, and 1 per cent iron oxide.

It has previously been shown by other investigators that calcination increases the activity of siliceous admixtures, probably by changing the material from a partly crystalline form to the more active amorphous state. Since volcanic ash has been calcined by nature, it is more than ordinarily active; however it was thought best to determine the effects of further calcination. Therefore, to investigate the effect of the condition of the silica, portions were calcined at various temperatures: (1) to incipient fusion at approximately 2200 F. (1200 C.), (2) to partial clinkering at approximately 2100 F. (1150 C.), and (3) to 1800 F. (980 C.). A fourth portion was not heated.

As the heating of the first two portions to 2200 and 2100 F. (1200 and 1150 C.), respectively, produced numerous coarse grains of fused silica, it was necessary to pregrind them before blending them with the cements. This was accomplished in a small batch mill until the fineness was such that 20 per cent was retained on a No. 200 sieve. The third portion, which was heated to 1800 F. (980 C.) and the fourth portion which was not heated were used as received.

The clinkers of the three portland cements were first ground in a batch mill until the specific surface (fineness as measured by surface area of particles) was 1300 sq. cm. per g. The time required to raise the fineness of the unblended portion from 1300 to 1500 sq. cm. per g. was carefully noted. The blends of silica and cement were then ground for this same length of time inasmuch as it was impossible to measure accurately the fineness of the blended cements. Various proportions of cement to either natural or calcined silica were used in the production of the blended cements, these proportions varying from 100 per cent of cement and no silica to 60 per cent of cement and 40 per cent of silica, by weight. For the blended cements produced by merely adding the silica to the cement, the three cements were first ground to a specific surface of 1500 sq. cm. per g.

## CONSISTENCY TESTS OF CONCRETE

As it is well known that the characteristics of the cement have an influence upon the water-cement ratio required to produce a given consistency, tests were made upon the several plain and blended cements to determine this relationship using a 1:5.2 concrete mix by weight and a 3-in. slump. The results of the tests are included at the bottom of Fig. 2. In general, it is seen that (1) the use of higher percentages of silica, at least with the normal-lime cement, requires the use of higher water-cement ratios, and (2) for cements blended with a given percentage of silica, the required water content varies inversely as the temperature of calcination.

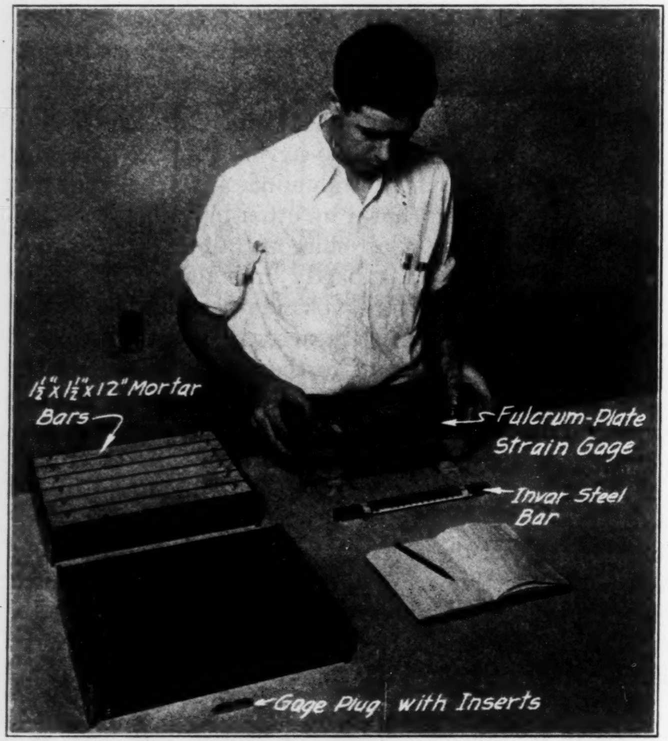
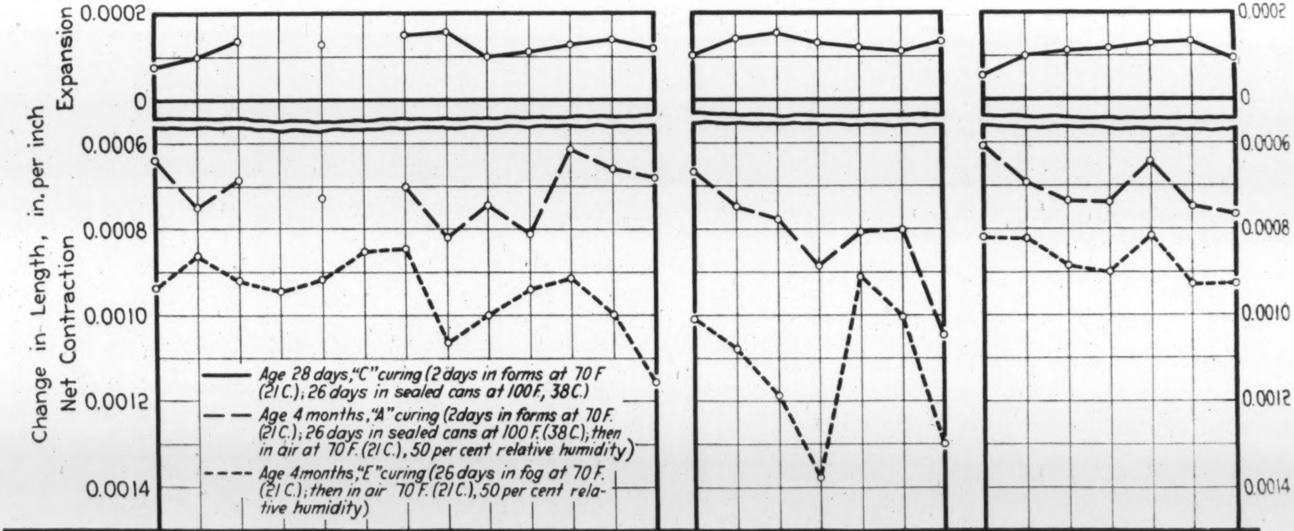


FIG. 1.—Apparatus for Molding and Measuring Volume Change Bars.



| | Normal-Lime Cement | | | | | | | | | | | | | Low-Lime Cement | | | | | | | High-Lime Cement | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Silica, per cent | 0 | 20 | 20 | 20 | 30 | 30 | 30 | 30 | 30 | 30 | 40 | 40 | 40 | 0 | 30 | 30 | 30 | 30 | 30 | 30 | 0 | 30 | 30 | 30 | 30 | 30 | 30 |
| Silica Calcination Temperature, deg. Fahr. | — | 2200 | 2100 | 1800 | 2200 | 2200 | 2100 | 1800 | — | — | 2200 | 2100 | 1800 | — | 2200 | 2100 | 1800 | 2200 | 2100 | 1800 | — | 2200 | 2100 | 1800 | 2200 | 2100 | 1800 |
| Silica Calcination Temperature, deg. Cent. | — | 1205 | 1150 | 980 | 1205 | 1205 | 1150 | 980 | — | — | 1205 | 1150 | 980 | — | 1205 | 1150 | 980 | 1205 | 1150 | 980 | — | 1205 | 1150 | 980 | 1205 | 1150 | 980 |
| Silica Added (Add.) or Interground (Gr.) | — | Gr. | Gr. | Gr. | Gr. | Add. | Add. | Add. | Gr. | Add. | Gr | Gr. | Gr. | — | Gr. | Gr. | Gr. | Add. | Add. | Add. | — | Gr. | Gr. | Gr. | Add. | Add. | Add. |
| Water-Cement Ratio by Weight | 0.546 | 0.553 | 0.549 | 0.546 | 0.574 | 0.549 | 0.560 | 0.586 | 0.561 | 0.583 | 0.559 | 0.588 | 0.608 | 0.574 | 0.570 | 0.575 | 0.580 | 0.567 | 0.587 | 0.622 | 0.536 | 0.558 | 0.558 | 0.582 | 0.567 | 0.561 | 0.581 |

FIG. 2.—Volume Changes of Plain and Silica-Blended Cement Mortars. Specimens are $1\frac{1}{2}$ by $1\frac{1}{2}$ by 12-in. mortar bars, cement-aggregate ratio $1:3\frac{1}{4}$ by weight using 0 to No. 4 sand.

## VOLUME CHANGE TESTS

*Molding and Curing Volume-Change Specimens:*

All volume-change specimens were made of one part cement to 3¼ parts sand by weight, the sand having a fineness modulus of 2.57. The water-cement ratio for each mortar was the same as that required to produce a 3-in. slump for corresponding concrete of 1:5.2 mix (see Fig. 2). The specimens for the volume-change tests are 1½ by 1½ by 12-in. mortar bars, with two brass plugs passing entirely through the bar on a 10-in. gage length. Stainless steel inserts in these brass plugs permit length changes on two opposite faces of the bars to be observed with a 10-in. fulcrum-plate strain gage. The molds, volume-change bar, stainless steel inserts and strain gage are shown in Fig. 1.

The volume-change bars were cured in groups, three bars for each condition, as shown in Table I.

TABLE I.—CURING CONDITIONS FOR VOLUME-CHANGE SPECIMENS.

| | Curing Conditions | |
|---|---|---|
| | 0 to 2 days (70 F., 21 C.) | After 2 days |
| Group A......... | Covered molds.. | 26 days in sealed can at 100 F. (38 C.); then in air at 70 F. (21 C.), 50 per cent relative humidity, without can. |
| Group C......... | Covered molds.. | 26 days in sealed can at 100 F. (38 C.); then under water at 70 F. (21 C.), without can. |
| Group D......... | Covered molds.. | 5 days in sealed can at 100 F. (38 C.); then in air at 70 F. (21 C.), 50 per cent relative humidity, without can. |
| Group E......... | Covered molds.. | 26 days without can in fog at 70 F. (21 C.); then in air at 70 F. (21 C.), 50 per cent relative humidity, without can. |

The first measurements on the volume-change specimens were made at the age of 2 days, at which age the molds were removed and the stainless steel inserts were placed in the brass plugs. While stored at 100 F. (38 C.), groups *A*, *C* and *D* were sealed in cans containing a few ounces of water.

*Volume-Change Test Results:*

The lengths of all volume-change bars were observed once each week for eight weeks, and thereafter have been observed once each month. Corrections are made for thermal expansion or contraction and, therefore, the data do not include the effect of temperature change.

The curing conditions following the preliminary curing period were chosen to represent the extremes of natural weather conditions, some specimens being placed in dry air (50 per cent relative humidity) and others being immersed in water. The drying condition is especially severe in view of the small size of the specimens. Some comparisons which have been made elsewhere indicate that the contraction of concrete in service would seldom approach the contraction of these mortar bars, even if correction is made for the difference in cement content. It remains to be determined whether or not the values obtained for the early periods of drying in these tests represent the results which might be expected for larger specimens in more humid atmosphere over long periods of time.

Values of the net contraction of mortars up to the age of 4 months are shown for two curing conditions in Fig. 2. The expansion for the first 28 days for a third curing condition is also included. The expansions of the specimens immersed in water after the age of 28 days are not included. However, the later expansions are in general only about 10 per cent greater than those at 28 days. Little importance is attached to mortar expansion in relation to siliceous materials as even though the effect of blended silica appears to be to increase expansion slightly, the total expansion is small in all cases.

The values of 4-month contractions plotted in Fig. 2 indicate at a glance that the preliminary curing condition of 100 F. (38 C.), simulating that of mass concrete, leads to less contraction than does curing continuously at 70 F. (21 C.). Furthermore, the difference in contraction for these two curing conditions appears to be about the same for all cements either plain or blended. Therefore, relative values can be discussed for both curing conditions simultaneously.

In examining the 4-month values of contraction in the graphs of Fig. 2, the most significant disclosure is the effect of silica, whether a part of the cement or added in the form of volcanic ash, in increasing the volume change. Comparing first the 3 plain cements, it is apparent that the lower the lime content, which is equivalent to stating the higher the silica content, the greater the contraction of the mortar. Similarly, comparing the cement-silica blends with the corresponding plain cements, it may be observed that, in general, the blended silica also increases the contraction. The effect of the blended silica apparently depends upon the lime (or silica) content of the cement with which it is blended, being large for the low-lime cement and small for either the high-lime or normal-lime cement. In fact, the blended silica affects contraction to such a slight degree when high-lime cement is used that some silica blends with this cement give less contraction than even the plain cements of either normal-lime or low-lime contents. Since the total silica content of the blends with the high-lime cement is 42 per cent, as compared with only 24 per cent for the plain low-lime cement, it is indicated that silica added as a granular material has less effect upon contraction than silica which is a chemical part of the cement.

The effect of the calcination temperature of the silica upon contraction of mortar may also be observed from the data of Fig. 2. In practically every case, the contraction of mortar containing a given percentage of blended silica is least for the highest temperature of calcination and greatest for the lowest temperature of calcination. The magnitude of the effect of temperature of calcination appears to be small for the blends of the high-lime cement but large for those of low-lime cement.

The effect upon contraction of mortar of intergrinding, as compared with merely mixing the silica and cement, is neither large nor consistent. For the high-lime cement, the interground blends show either the same or slightly less contraction. Inasmuch as the relative fineness must be a factor in these comparisons, it is not believed that the data warrant more than the statement that the effect of intergrinding is not large.

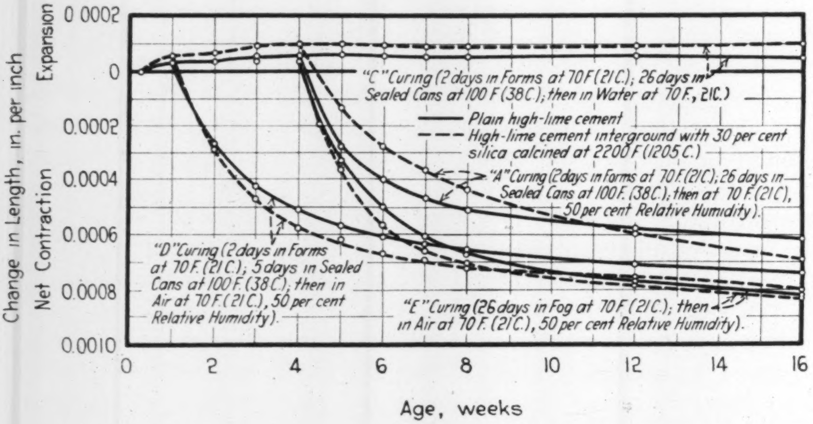


Fig. 3.—Effect of Age and Curing Conditions on Volume Changes of Plain and Silica-Blended Cement Mortar.

Figure 3 shows the progressive volume changes with time under various curing conditions of mortar bars containing high-lime cement, both plain and blended with 30 per cent of silica. It may be seen that the cement-silica blend exhibits less contraction during the early periods of drying but greater contraction after long periods of drying for all conditions of curing. This is true for the normal-lime and low-lime cements as well.

The effect of preliminary curing upon the subsequent contraction of mortar is shown by the curves in Fig. 3. Greater contraction is obtained for continuous curing at 70 F. (21 C.) than for preliminary curing for 28 days at 100 F. (38 C.), and this difference at the age of 4 months amounts to more than 20 per cent for the blended cement and more than 30 per cent for the plain cement. The difference for the plain cement seems to be remaining constant at this age while the difference for the blended cement seems to be diminishing rapidly. This is due to the pronounced effect of preliminary curing at 100 F. (38 C.) in delaying the contraction of the blended cement. In fact, the contraction of the blended-cement mortar for this preliminary curing occurs so slowly after exposure to dry air that the contraction at early ages is less than half that of the plain-cement mortar.

## COMPRESSIVE-STRENGTH TESTS

### *Molding and Curing Compression Specimens:*

The compression cylinders were made of the same mortar mix, one part cement to 3¼ parts graded sand, as were the volume-change bars, and were cast in 2 by 4-in. metal cans. Previous tests had indicated that the effect of blending cements upon the compressive strength of mortar is different from the effect for concrete. In a limited series of tests, with the silica here used but with a regular commercial cement, maximum aggregate sizes were varied from ⅛ in. to 3 in. It was shown that the relative strength for blended cement as compared with plain cement was greater for aggregates of greater maximum size. For the present study, however, mortar was selected because it was believed that the variables of silica calcination, curing conditions, chemical composition of cement and method of blending the silica could be satisfactorily investigated without the expense of large specimens. It should, therefore, be borne in mind that these tests are made on mortars and not on concretes.

TABLE II.—CURING CONDITIONS FOR COMPRESSION SPECIMENS.

| | Curing Conditions | | | | |
|---|---|---|---|---|---|
| | 0 to 2 days (70 F., 21 C.) | 2 to 28 days | | After 28 days (70 F., 21 C.) | |
| | Container | Container | Temperature | Container | Relative Humidity |
| Group A | Sealed | Sealed | 100 F. (38 C.) | None | 50 per cent |
| Group B | Sealed | Sealed | 100 F. (38 C.) | Sealed | ...... |
| Group C | Sealed | Sealed | 100 F. (38 C.) | None | Under water |
| Group E | Unsealed (fog) | None (fog) | 70 F. (21 C.) | None | 50 per cent |

The compression cylinders that were maintained constantly at 70 F. (21 C.) were stripped of their molds at the age of 2 days; all others were sealed in their metal containers and stored for at least 28 days. The curing conditions of the various groups of specimens are shown in Table II.

TABLE III.—COMPRESSIVE STRENGTH OF PLAIN-CEMENT AND BLENDED-CEMENT MORTARS UP TO THE AGE OF SIX MONTHS.

| Cement, per cent | Silica, per cent | Calcination Temperature of Silica | | Curing Conditions[a] | | | | Compressive Strength, lb. per sq. in. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | deg. Fahr. | deg. Cent. | Group | 2 to 28 days | | After 28 days (70 F., 21 C.) | 7 days | 28 days | 3 months | 6 months |
| | | | | | deg. Fahr. | deg. Cent. | | | | | |
| NORMAL-LIME CEMENT | | | | | | | | | | | |
| 100 | 0 | .... | .... | A | 100 | 38 | 50% R. H.[b] | 3860 | 5350 | 6110 | 6570 |
| 100 | 0 | .... | .... | B | 100 | 38 | Sealed can | 3860 | 5350 | 5690 | 5780 |
| 100 | 0 | .... | .... | C | 100 | 38 | Water | 3860 | 5350 | 5230 | 5430 |
| 100 | 0 | .... | .... | E | 70 | 21 | 50% R. H. | 2830 | 4030 | 6160 | 5860 |
| 80 | 20 | 2200 | 1205 | A | 100 | 38 | 50% R. H. | 2370 | 4790 | 5920 | 5160 |
| 80 | 20 | 2200 | 1205 | B | 100 | 38 | Sealed can | 2370 | 4790 | 4700 | 4660 |
| 80 | 20 | 2200 | 1205 | C | 100 | 38 | Water | 2370 | 4790 | 4280 | 4950 |
| 80 | 20 | 2200 | 1205 | E | 70 | 21 | 50% R. H | 2320 | 2910 | 4740 | 4020 |
| 80 | 20 | 2100 | 1150 | A | 100 | 38 | 50% R. H. | 2600 | 4420 | 5620 | 5460 |
| 80 | 20 | 2100 | 1150 | B | 100 | 38 | Sealed can | 2600 | 4420 | 4790 | 4970 |
| 80 | 20 | 2100 | 1150 | C | 100 | 38 | Water | 2600 | 4420 | 4960 | 5020 |
| 80 | 20 | 1800 | 980 | A | 100 | 38 | 50% R. H. | 2710 | 4580 | 5300 | 5460 |
| 80 | 20 | 1800 | 980 | B | 100 | 38 | Sealed can | 2710 | 4580 | 4530 | 4880 |
| 80 | 20 | 1800 | 980 | C | 100 | 38 | Water | 2710 | 4580 | 4550 | 4590 |
| 70 | 30 | 2200 | 1205 | A | 100 | 38 | 50% R. H. | 2350 | 3970 | 5080 | 4900 |
| 70 | 30 | 2200 | 1205 | B | 100 | 38 | Sealed can | 2350 | 3970 | 4130 | 4630 |
| 70 | 30 | 2200 | 1205 | C | 100 | 38 | Water | 2350 | 3970 | 3780 | 3870 |
| 70 | 30 | 2200 | 1205 | E | 70 | 21 | 50% R. H. | 1870 | 3030 | 3090 | 3640 |
| 70 | 30[c] | 1800 | 980 | A | 100 | 38 | 50% R. H. | 1830 | 3510 | 4520 | 4470 |
| 70 | 30[c] | 1800 | 980 | B | 100 | 38 | Sealed can | 1830 | 3510 | 4080 | 3950 |
| 70 | 30[c] | 1800 | 980 | C | 100 | 38 | Water | 1830 | 3510 | 3680 | 3980 |
| 70 | 30 | .... | .... | A | 100 | 38 | 50% R. H. | 2570 | 4870 | 5470 | 4930 |
| 70 | 30 | .... | .... | C | 100 | 38 | Water | 2570 | 4870 | 5140 | 4830 |
| 60 | 40 | 2200 | 1205 | A | 100 | 38 | 50% R. H. | 1870 | 3650 | 4260 | 4590 |
| 60 | 40 | 2200 | 1205 | B | 100 | 38 | Sealed can | 1870 | 3650 | 3850 | 4100 |
| 60 | 40 | 2200 | 1205 | C | 100 | 38 | Water | 1870 | 3650 | 3770 | 3830 |
| 60 | 40 | 2200 | 1205 | E | 70 | 21 | 50% R. H. | 1480 | 2330 | 3610 | 3200 |
| 60 | 40 | 2100 | 1150 | A | 100 | 38 | 50% R. H. | 1830 | 3490 | 3960 | 4190 |
| 60 | 40 | 2100 | 1150 | B | 100 | 38 | Sealed can | 1830 | 3490 | 3540 | 3760 |
| 60 | 40 | 2100 | 1150 | C | 100 | 38 | Water | 1830 | 3490 | 3420 | 4010 |
| 60 | 40 | 1800 | 980 | A | 100 | 38 | 50% R. H. | 1670 | 3840 | 3980 | 4180 |
| 60 | 40 | 1800 | 980 | B | 100 | 38 | Sealed can | 1670 | 3870 | 3690 | 3770 |
| 60 | 40 | 1800 | 980 | C | 100 | 38 | Water | 1670 | 3870 | 3720 | 3850 |
| LOW-LIME CEMENT | | | | | | | | | | | |
| 100 | 0 | .... | .... | A | 100 | 38 | 50% R. H. | 2250 | 4270 | 5260 | 5100 |
| 100 | 0 | .... | .... | B | 100 | 38 | Sealed can | 2250 | 4270 | 4820 | 4940 |
| 100 | 0 | .... | .... | C | 100 | 38 | Water | 2250 | 4270 | 4250 | 5140 |
| 100 | 0 | .... | .... | E | 70 | 21 | 50% R. H. | 1750 | 3470 | 4520 | 3990 |
| 70 | 30 | 2200 | 1205 | A | 100 | 38 | 50% R. H. | 1400 | 3260 | 3890 | 4020 |
| 70 | 30 | 2200 | 1205 | B | 100 | 38 | Sealed can | 1400 | 3260 | 3330 | 3500 |
| 70 | 30 | 2200 | 1205 | C | 100 | 38 | Water | 1400 | 3260 | 3380 | 3470 |
| 70 | 30 | 2200 | 1205 | E | 70 | 21 | 50% R. H. | 1090 | 1870 | 3220 | 2820 |
| 70 | 30 | 2100 | 1150 | A | 100 | 38 | 50% R. H. | 1600 | 3550 | 4040 | 4260 |
| 70 | 30 | 2100 | 1150 | C | 100 | 38 | Water | 1600 | 3550 | 3500 | 3600 |
| 70 | 30 | 1800 | 980 | A | 100 | 38 | 50% R. H. | 1400 | 3200 | 3590 | 3800 |
| 70 | 30 | 1800 | 980 | C | 100 | 38 | Water | 1400 | 3200 | 3120 | 3570 |
| 70 | 30[c] | 1800 | 980 | A | 100 | 38 | 50% R. H. | 890 | 2440 | 3130 | 2970 |
| 70 | 30[c] | 1800 | 980 | C | 100 | 38 | Water | 890 | 2440 | 2740 | 2780 |
| HIGH-LIME CEMENT | | | | | | | | | | | |
| 100 | 0 | .... | .... | A | 100 | 38 | 50% R. H. | 3670 | 4510 | 5180 | 5360 |
| 100 | 0 | .... | .... | C | 100 | 38 | Water | 3670 | 4510 | 4560 | 5040 |
| 100 | 0 | .... | .... | E | 70 | 21 | 50% R. H. | 3060 | 4350 | 5980 | 5060 |
| 70 | 30 | 2200 | 1205 | A | 100 | 38 | 50% R. H. | 2100 | 3970 | 5230 | 5140 |
| 70 | 30 | 2200 | 1205 | C | 100 | 38 | Water | 2100 | 3970 | 4220 | 4360 |
| 70 | 30 | 2200 | 1205 | E | 70 | 21 | 50% R. H. | 1900 | 3220 | 4090 | 3880 |
| 70 | 30 | 2100 | 1150 | A | 100 | 38 | 50% R. H. | 2620 | 4610 | 4830 | 5610 |
| 70 | 30 | 2100 | 1150 | C | 100 | 38 | Water | 2620 | 4610 | 4200 | 4150 |
| 70 | 30 | 1800 | 980 | A | 100 | 38 | 50% R. H. | 2380 | 4290 | 5410 | 5120 |
| 70 | 30 | 1800 | 980 | C | 100 | 38 | Water | 2380 | 4290 | 4530 | 4570 |
| 70 | 30[c] | 1800 | 980 | A | 100 | 38 | 50% R. H. | 2220 | 3780 | 4920 | 4660 |
| 70 | 30[c] | 1800 | 980 | C | 100 | 38 | Water | 2220 | 3780 | 3980 | 4130 |

[a] Groups *A*, *B* and *C* cured 2 days at 70 F. (21 C.), 26 days at 100 F. (38 C.) in sealed cans; thereafter at 70 F (21 C.) as shown. Group *E* cured in fog at 70 F. (21 C.) first 28 days.

[b] R. H. = relative humidity.

[c] Silica added without intergrinding. All other blended cements, silica interground with cement.

*Compression Test Results:*

The detailed results of the compression tests up to the age of 6 months are presented in Table III. A summary of the results at the age of 6 months is given in Table IV, in which all strengths are reported as percentages of the compressive strengths of similarly cured mortars containing unblended cement.

TABLE IV.—RELATIVE COMPRESSIVE STRENGTHS OF PLAIN-CEMENT AND BLENDED-CEMENT MORTARS AT THE AGE OF SIX MONTHS.

| | Silica, per cent | Calcination Temperature of Silica | | Relative Compressive Strength, per cent of plain-cement mortar | | | | |
|---|---|---|---|---|---|---|---|---|
| | | deg. Fahr. | deg. Cent. | Group A Curing[a] | Group B Curing[a] | Group C Curing[a] | Group E Curing[a] | Average of Groups A and C Curing |
| **NORMAL-LIME CEMENT** | | | | | | | | |
| Series 1 | 20 | 1800 | 980 | 83 | 84 | 85 | .. | |
| Series 2 | 20 | 2100 | 1150 | 83 | 86 | 93 | .. | |
| Series 3 | 20 | 2200 | 1205 | 79 | 81 | 91 | 69 | |
| Average | | | | 82 | 84 | 90 | | 86 |
| Series 4 | 30 | .... | .... | 75 | .. | 89 | .. | |
| Series 5 | 30[b] | 1800 | 980 | 68 | 68 | 73 | .. | |
| Series 6 | 30 | 2200 | 1205 | 75 | 80 | 71 | 62 | |
| Average | | | | 73 | | 78 | | 76 |
| Series 7 | 40 | 1800 | 980 | 64 | 65 | 71 | .. | |
| Series 8 | 40 | 2100 | 1150 | 64 | 65 | 74 | .. | |
| Series 9 | 40 | 2200 | 1205 | 70 | 71 | 71 | 55 | |
| Average | | | | 66 | 67 | 72 | | 69 |
| **LOW-LIME CEMENT** | | | | | | | | |
| Series 10 | 30[b] | 1800 | 980 | 58 | .. | 54 | .. | |
| Series 11 | 30 | 1800 | 980 | 75 | .. | 70 | .. | |
| Series 12 | 30 | 2100 | 1150 | 84 | .. | 70 | .. | |
| Series 13 | 30 | 2200 | 1205 | 79 | 71 | 68 | 71 | |
| Average | | | | 74 | | 65 | | 70 |
| **HIGH-LIME CEMENT** | | | | | | | | |
| Series 14 | 30[b] | 1800 | 980 | 87 | .. | 82 | .. | |
| Series 15 | 30 | 1800 | 980 | 96 | .. | 91 | .. | |
| Series 16 | 30 | 2100 | 1150 | 105 | .. | 82 | .. | |
| Series 17 | 30 | 2200 | 1205 | 96 | .. | 86 | 77 | |
| Average | | | | 96 | | 85 | | 91 |

[a] See Table II for curing conditions.
[b] Added without intergrinding.

Considering the 7-day compressive strengths shown in Table III, it is seen in general that for all curing conditions the effect of blending cement with silica is to reduce the strength of the mortar, and that the percentage of this reduction in strength is larger than the percentage of reduction in amount of the cement. It is thus indicated that during the first 7 days the silica has contributed little or nothing to the strength.

The 28-day compressive strengths shown in Table III, however, show a different relation, especially for the 100 F. (38 C.) preliminary curing conditions. The cement-silica blends at this age exhibit strengths which are relatively higher than at 7 days, indicating that the silica is contributing to the strength. At still later ages, the effect is more pronounced. The degree to which the silica contributes to strength apparently depends upon the chemical composition of the cement with which it is blended, since for the high-lime cement the strengths for the blends are almost as high as those for the plain cement, while for the low-lime cement the strengths for the blends indicate only a slight contribution from the blended silica.

For the normal-lime cement, the only one with which different percentages of silica were blended, it is evident that increasing the amount of silica results in lower mortar strength regardless of the calcination of the silica. Considering the average 6-month strengths under the two curing conditions, groups *A* and *C* for which data are available for all percentages of silica, the compressive strengths for mortars using 20, 30, and 40 per cent silica are respectively 14, 24, and 31 per cent lower than that for the plain cement. Thus for the normal-lime cement apparently insufficient lime is liberated up to the age of 6 months to combine with even the smallest amount of silica employed in these tests, 20 per cent.

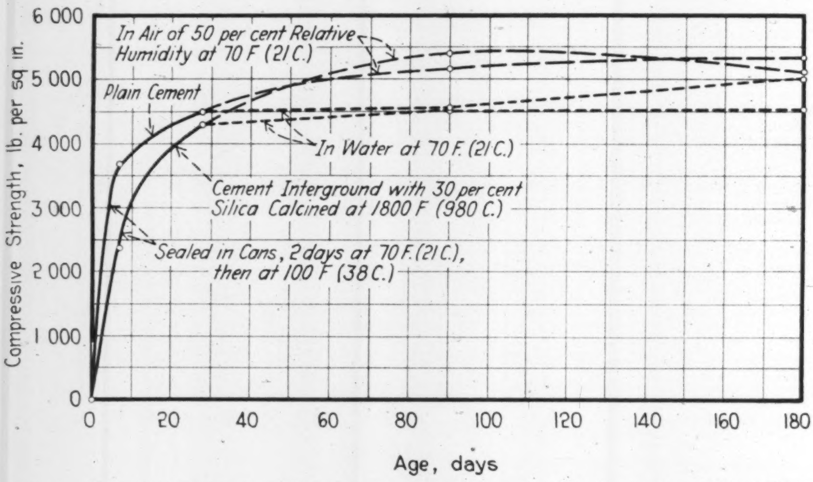


FIG. 4.—Effect of Interground Silica upon the Compressive Strength of High-Lime Cement Mortar.

Some of the more favorable test results with regard to the effect upon strength of blending silica with cement are shown in Fig. 4, for high-lime cement blended with 30 per cent of silica which had been calcined at 1800 F. (980 C.). For the specimens stored in air after the age of 28 days, it is seen that the strength of the silica blend is about equal to that of the plain cement. The fact that the gain in strength of the specimens stored in water after 28 days is small at the age of 3 months is most probably due to a difference in the condition of test. The 28-day specimens were fairly dry when tested while the 3-month specimens were thoroughly saturated.

A comparison of the strengths obtained by the use of silica calcined at various temperatures, as presented in Table IV, shows that on the average but little benefit to strength is gained by calcination. As pointed out previously, the type of silica employed is fairly active without calcination. It appears that the strengths of mortars containing cements interground with silica are higher than for the blended cements in which the silica is merely mixed with the cement.

## HEAT-GENERATION TESTS

An important consideration for massive concrete construction is the extent, if any, to which heat of hydration can be reduced by substituting siliceous materials for a portion of the cement. It is now fairly well established that the heat of hydration of regular portland cement can be varied within certain limits by varying the chemical composition. However, even the lowest heat of hydration consistent with other desirable properties of the concrete is sufficient to necessitate either artificial or natural joints. Therefore, further reduction in heat of hydration than is obtainable by altering only the chemical composition of the cement would be advantageous.

To investigate properly the effect of silica upon heat of hydration, several factors must be considered. The all-round performance of a cement-silica blend seems to be best when a high-lime cement is used. On the other hand, an engineer would not advocate the use of a high-lime cement for mass concrete if the cement were to be used plain. It seems logical that the properties of the best cement-silica blend should be compared with those of the best plain cement rather than those of the cement used in making the blend. In this connection, it is believed that strength alone should not be given too much consideration, since strengths far above the requirements are usually obtained when the cement content is fixed at the minimum necessary to maintain other properties at safe levels. Therefore, attention should be directed particularly toward comparing a low-lime cement which is considered to be best suited to mass concrete with, first, a mixture of silica with a high-lime cement and, second, a mixture of the silica with the low-lime cement.

### *Apparatus and Methods of Test:*

In this investigation, the heat of hydration of cement-silica mixtures could not be obtained accurately from a heat-of-solution calorimeter because free silica is not readily soluble in acids. Furthermore, the cost of making a large number of tests with adiabatic calorimeters employing large concrete cylinders was prohibitive. Therefore, a new type of calorimeter, called the "vane calorimeter," was developed which permits measurement of heats of hydration with fair accuracy at a moderate cost.

The vane calorimeter takes advantage of the high concentration of cement in a neat paste, recognizing the fact that cement in a paste generates the same amount of heat as the same cement in concrete only when water-cement ratios and curing conditions are identical. Comparative tests indicated that for low-heat cements a constant curing temperature of 100 F. (38 C.) gives heat-of-hydration results approximately equal to those obtained under the variable temperature conditions naturally occurring in large masses of concrete cast at 70 F. (21 C.). Therefore, specimens for these tests are cured in a room having the constant temperature of 100 F. (38 C.). Furthermore, comparative tests had shown that reliable results could be obtained with a constant water-cement ratio of 0.40 by weight, there being a small and consistent difference between the results obtained with this water-cement ratio and with the higher water-cement ratios employed for concrete. Therefore, the constant water-cement ratio of 0.40 by weight was used for all heat-of-hydration tests. Both the curing temperature and the water-cement ratio thus employed are similar to those specified for acceptance tests on cement for the Boulder Dam.

The neat-cement specimen for the heat-of-hydration test, weighing about four pounds, is placed in an interior compartment of the vane calorimeter. The heat which is generated in the specimen is conducted to the outer surface of the calorimeter along metal vanes symmetrically spaced. The temperature of the specimen rises a few degrees above that of the room during the first day and thereafter is within a degree or two of the room temperature. The temperature difference between the inner compartment and the outer surface, which is of heavy copper protected from fluctuations in room temperature by an insulating shield, is proportional to the rate at which heat is being removed from the specimen. The constant of proportionality is determined in a separate test by observing the temperature difference when a known amount of electrical heat is released in the interior compartment. The temperature difference is determined to an accuracy of about 0.02 F. (0.01 C.) by measuring the electrical resistance ratio of two thin flat copper coils, one attached to the interior compartment and one attached to the outer surface. This accuracy is readily obtained since the resistance ratio varies only slightly from unity, which is a favorable condition for accurate measurement. The rates of heat liberation for each hour, expressed in calories per hour and obtained from the temperature differences at various ages, are summed up to obtain the cumulative totals for any desired period of hydration. After the first two days larger units of time are chosen for the summation.

*Heat-of-Hydration Test Results:*

Heat of hydration tests up to the age of 28 days for cements blended with silica calcined at 2100 F. (1150 C.) are given in Table V.

It is apparent that the blending of siliceous material with cement causes a reduction in heat of hydration by almost the full percentage of cement replacement for low-lime and normal-lime cements. In the case of the high-lime cement the greater reaction of the siliceous material is evidenced by the fact that replacing 30 per cent of cement by silica causes a reduction in 28-day heat of hydration of 20 per cent, which is two-thirds the amount of cement replacement. Since the compressive strengths of the blended high-lime cement mortars are reduced a negligible amount, it is indicated that the heat of hydration is reduced more than is strength. A most instructive comparison can be made between heat-of-hydration values for high-lime cement containing 30 per cent of siliceous material and plain low-lime cement, for which cements the mortar strengths are nearly equal. It may be observed that the 28-day heat of hydration of the high-lime cement blended with silica is 74 cal. per g. as compared with 79 cal. per g. for the low-lime cement. Since the low-lime cement contained 7 per cent tricalcium aluminate while the high-lime cement contained practically none, it is believed that the heats

TABLE V.—HEAT OF HYDRATION OF PLAIN AND BLENDED CEMENTS.

| | Cement, per cent | Silica, per cent | Heat of Hydration, cal. per g. | | |
|---|---|---|---|---|---|
| | | | 3 days | 7 days | 28 days |
| Normal-lime cement | 100 | 0 | 74.3 | 83.5 | 93.5 |
| | 70 | 30 | 51.7 | 60.1 | 69.2 |
| | 70 | 30[a] | 50.5 | 58.9 | 66.9 |
| Low-lime cement | 100 | 0 | 58.5 | 66.7 | 78.9 |
| | 70 | 30 | 42.8 | 49.3 | 58.9 |
| High-lime cement[b] | 100 | 0 | 69.4 | 81.1 | 92.6 |
| | 70 | 30 | 52.0 | 61.6 | 74.0 |

[a] Silica added without intergrinding. All other blended cements, silica interground with cement.

[b] This cement contained practically no tricalcium aluminate, while the normal-lime cement contained 6 per cent and the low-lime cement contained 7 per cent.

of hydration would be practically equal for plain low-lime cement and blended high-lime cement of equal tricalcium aluminate content.

## CONCLUSIONS

The conclusions here presented are based upon the results of the limited number of tests completed to date and are purely tentative, since later test results may alter the opinions herein expressed. It should be emphasized that the conclusions apply only to three types of portland cement, one type of silica (volcanic ash), four conditions of treatment with regard to the preliminary calcination of the silica, and five arbitrary sequences of curing and storage conditions. The tests herein described are to be continued, and durability tests will be made. Nevertheless, the tests so far made show promise in the use of a high-lime cement blended with silica, especially for mass concrete.

Also it is evident that blended high-lime cements can be produced more economically than plain low-lime cements, at least if adequate supplies of cheap silica are available, due to (1) a reduction in amount of clinker required and (2) the well-known fact that high-lime cement is more easily ground than low-lime cement.

In order to investigate more fully the possibilities in the blending of silica and cement and to determine the most favorable composition, it is believed that further studies are justified along the following lines: (*a*) use of different types of silica, (*b*) further variations in curing conditions, especially to simulate normal weather, (*c*) use of concrete instead of mortar specimens, (*d*) effect of exposure to sea water, and (*e*) relative manufacturing costs.

The tentative conclusions follow:

1. The contraction of mortar specimens upon drying is less at the early ages and greater at the later ages for cement-silica mixtures than for plain cement.

2. Contractions of mortar up to the age of 4 months are in many cases less for blends of high-lime cement with silica than for plain cement of normal-lime or of low-lime content.

3. The calcination of the silica has an appreciable effect upon the contraction of mortars containing blended cements; the higher the temperature of calcination, the less the contraction.

4. The expansion of mortar during a period of immersion in water is slightly greater for blended cement than for plain cement.

5. In general the blending of active silica with cement tends to lower the compressive strength of mortars up to the age of 6 months.

The amount of this reduction in strength is governed largely by the chemical composition of the cement.

6. At least 30 per cent of a high-lime cement can be replaced by interground silica without appreciable reduction in the compressive strength at the ages of 3 or 6 months.

7. Higher compressive strength is obtained if silica is interground with cement than if silica is merely mixed with cement, at least up to the age of 6 months.

8. The heat of hydration of a blend of cement and silica is less than that of a corresponding plain cement, but the reduction is not proportionately as great as the percentage of replaced cement.

9. The heats of hydration of plain low-lime cement and of high-lime cement blended with 30 per cent silica, are practically equal.

*Acknowledgment.*—The authors are indebted to J. W. Fry, J. A. Jameson, and R. J. Villagrana, senior and graduate students in civil engineering, who conducted most of the tests included in this investigation.

# DISCUSSION

Mr. P. H. Bates[1] (*presented in written form*).—Due to the wide variety of siliceous materials, both of natural and artificial origin, it would have been much better had the authors been a little more specific in their title and used the expression "volcanic ash" instead of "siliceous material." The material blended with the cements was a volcanic ash containing by analysis 75 per cent $SiO_2$. The partial analysis would indicate that possibly as much as 50 per cent of this ash was one of the very many forms of silica. Sossman in his book on "The Properties of Silica," makes the statement, "One of the most striking facts about silica is the variety of aspects under which it occurs." He then cites eight better known modifications (low quartz, high quartz, low tridymite, lower high tridymite, upper high tridymite, low crystobalite, high crystobalite, and vitreous silica) and two other *groups* of forms of silica, namely, chalcedonic and amphorous variety. Since all of these forms of silica have different physical and chemical properties, it is rather unfortunate that the authors chose to use such a word as "silica" to describe the material with which they were working.

It is suggested that in any further work microscopic and X-ray examinations be made of the ash to determine just what form of silica is under consideration, and, furthermore, that after the heat treatments such examination be made without question. The various forms of silica are very apt to change into different forms under heat treatment.

In the early part of the paper the authors make the statement that cement of low lime content is considerably more difficult to grind than that of higher lime content. This statement is questioned. As these two types of cement would be produced now, it is possibly correct. On the other hand, however, high-lime cements as now burned are coke-like materials. The low-lime cements as now burned are dense, almost vitrified, products. It is quite probable that if cement burners would realize the two types of cement need not be burned at the same temperatures and would therefore burn only to the same physical condition of the clinker, there would be no difference in the grindability of the two types of cement; further, in blending the raw mix for the two types, it also would not be necessary to use as much fluxing material in the low-lime cements as in the high lime, but, unfortunately, at the present time, the tendency seems to be towards using rather more flux in the low-lime cements than in the high-lime cements, thus, again, tending to produce a much harder clinker, which is entirely unnecessary if proper mixes were used.

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[1] Chief, Clay and Silicate Products Division, U. S. Bureau of Standards, Washington, D. C.

MR. W. C. HANNA.[1]—I wish to call attention to the fact that the authors have carried the tests for only very short periods and that the results possibly would be entirely different if they could carry them forward for long periods, say ten or fifteen years. I have made a great many tests on different adulterated cements which seemed to be very promising at short periods, even as much as two years, but after possibly ten or fifteen years, the results would be entirely different. I was recently surprised to note from some of our tests, which covered about fifteen years, on adulterated cements of this type, that some which showed up better than normal portland cement up to periods of about two years, were only about half what portland cement was at the age of ten or fifteen years. I believe where this type of adulterated cement has been used in practice it has proved to be unsatisfactory.

MR. F. O. ANDEREGG.[2]—It would be of great interest to run the analysis for active silica as carried out by European investigators.[3] Recently Feret[3] has reported a series of experiments involving European pozzuolanic materials added as admixtures, to the extent of about 30 per cent of the weight of the cement. The great increase in the water requirement seriously affected the strengths, but correcting for this, evidence was obtained of some combination resulting in added strength. Feret concluded that more than 3 per cent could hardly be added in practise, too small an amount to have appreciable pozzuolanic action. In another article,[4] he states that a cement made by grinding two parts of portland cement with about one of a French pozzuolan, Gaize, gave very satisfactory results when aided by temperatures such as are encountered in the French Colonies. His results are quite similar to those reported by Carlson and Troxell.

The reason that Carlson and Troxell found more action when the tricalcium silicate content of the cement was high was, first, because of the extra amount of reactive lime set free, and, second, because the reaction rate is very sensitive to temperature, and the greater amount of heat set free on hydrating tricalcium silicate would accelerate the reaction.

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[1] Chemist, California Portland Cement Co., Colton, Calif.

[2] Consulting Specialist on Building Materials, Pittsburgh, Pa.

[3] R. Feret, "Nature and Progress of the Pozzuolanic Reaction," *Revue des Matériaux de Construction et de Travaux Publics*, No. 281, p. 41 (1933); No. 282, p. 93 (1933).

[4] R. Feret, "Colonial Concretes," *Revue des Matériaux de Construction et de Travaux Publics*, No. 283, p. 157 (1933).

MR. R. S. GREENMAN.[1]—I have been interested in the use of cement since 1900—thirty-three years. In the early nineteen hundreds there were quite a few attempts made to adulterate cement. About that time there was produced a silica cement which was put in use in a dam upon the Hudson River; and when this dam was inspected in 1917 it was a fine, sound, hard concrete, with no evidences of seepage or carbonate of lime deposits on the face, and it gave an excellent demonstration of that earlier adulterated cement product. It has since been over-coated because of a raise in the height of the dam. I believe that this paper, which is a scientific study of the question of adulterated cement, should be taken into careful consideration in connection with present-day investigations that are being made by the chemists of various companies on the beneficial effect of more silica.

MR. M. A. SWAYZE.[2]—The authors of this paper refer to the large amounts of hydrated lime produced during the hydration of portland cement, and reference was also made to the fact that this hydrated lime was soluble. The popular conception seems to be that concrete made from portland cement is a perfectly pervious body through which water can circulate at will and dissolve therefrom the hydrated lime set free by the hydration of the tricalcium silicate component of the cement, and thereby weaken the structure. That attitude is based on wrong premises, first of which is the assumption that concrete is a perfectly permeable body through which water can circulate. There is no great difference between cements of the ordinary composition as we have them today, and the high-lime cements which have been produced for the past seven or eight years. The difference in the amount of hydrated lime set free by the hydration of tricalcium silicate, first of all, is a rather minor difference. The average portland cement will liberate 12 to 15 per cent of hydrated lime during its hydration. The high-early-strength cements which are produced by increasing the tricalcium silicate content in very few instances will liberate, when completely hydrated, more than 20 or 21 per cent. Now if concretes permeable to water are made with either type of cement, we should not expect those concretes to be permanent. But in *properly* designed concrete, we find in every instance the proportions are such that that concrete will be *impermeable.* Therefore in the solution of lime from that concrete, we are only concerned with the surface of that concrete; in other words, surface attack. In this matter, it seems to me that those who are concerned about hydrated lime are only carrying their chemistry half far enough; they know that tricalcium silicate hydrates, and that hydrated lime is set free during that reaction, but they neglect to consider that in any natural exposure we always have carbon dioxide present, and the moment it comes in contact with hydrated lime it produces calcium carbonate, which is a very insoluble material.

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[1] Consulting Concrete Engineer, Albany, N. Y.

[2] Chief Chemist, Lone Star Cement Co. New York, Inc., Hudson, N. Y.

MR. ANDEREGG.—Of the sixteen or twenty factors which affect the rate of attack of corrosive waters on concrete, the lime content of the cement might be ranked about number eight or ten. The most important factor is the opportunity the aggressive water may have to come in contact with the lime in the concrete, which depends upon the density of the concrete. Therefore, a high-lime cement, ground properly so as to give a very compact, dense paste, will be found more resistant to corrosive agencies than a standard portland cement, other things being equal.

MR. T. C. POWERS.[1]—We should not lose sight of the fact that the data presented represent results from one of the most active of the available silicas. Mr. Anderegg pointed out that experiments by Feret in Europe show very negative results. I think you will find, if you survey the literature, a great deal of contradictory test results reported by different investigators. The reasons for these contradictions are very many, too many to be discussed in detail, except to say that the great difference in materials is one very important factor contributing to such contradictory results. The particular material discussed in the paper is one of the most active so that when discussing this material we are considering pozzuolanas in their best possible light.

Mr. Greenman stated that the data of this report indicated the superiority of a high silica content. I do not believe that the authors of the paper themselves will agree completely with that interpretation. Volume change was increased by the use of silica and no increase in strength was attained which could not have been effected by the use of straight portland cement mixes. Our tests have shown that it is always possible to produce a higher strength with straight portland cement than with any blend, simply because a given quantity of portland cement is basically more cementitious than an equal quantity of a blend; that is, of cement diluted with some less active material.

Another point should be given much emphasis—that it is not only the chemical activity of the added silica but also its effect upon the availability of the soluble constituents of the cement which determines the effectiveness of the silica. When a powdered material is added to a mix it is necessary, as a rule, to increase the water content. This added water increases the porosity of the cementing paste and thus makes the soluble constituents of the cement more easily available to solvents. The increase in porosity is, of course, detrimental and competes against any beneficial effect that silica may have. For any given material these two effects strike a balance at some quantity of added silica. With some materials that balance may occur at a relatively high percentage, but usually, as Mr. Anderegg pointed out, at a rather low percentage, around 2 to 3 per cent with many materials. Such a small quantity can have no important chemical effect; its benefits, if any, are chiefly physical.

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[1] Associate Engineer, Portland Cement Assn., Chicago, Ill.

MR. J. C. PEARSON.[1]—I should like to ask one question, because in planning an investigation of this sort, one would naturally consider that one of the major variables would be the fineness of the admixture of siliceous material. Was any consideration given to that in this particular case?

THE CHAIRMAN (*Mr. R. E. Davis*[2]).—An attempt was made to secure the same degree of fineness for the cement with which silica was ground, as the cement to which no silica was added; the specific surface for each cement was about 1500 sq. cm. per g. There is always the question as to how fine the silica really was after the grinding operation was completed. We have no way of separating the silica particles from the rest after the cement-silica mixture has been ground.

MR. C. H. SCHOLER.[3]—I noticed in Fig. 4 a retrogression in strength with increased age on the part of the blended cement as compared with portland cement. That retrogression apparently was sufficient to cause it to show lower strength than unblended portland cement. I should like to inquire whether the authors attach any significance to this fact?

With reference to the plasticity of the mortar and the water required to give it the same plasticity, would there be enough difference in water content to account for the difference in strength shown by the two types of cements?

MR. C. P. DERLETH[4] (*presented in written form*).—The volcanic ash used in the authors' series is designated as silica probably for the reason that it contains approximately 75 per cent silica. The impression resulting is that only the active silica content is reactive with lime. Nagai[1] and the European authorities, Kuhl, Michaelis, and others have found that the aluminous constituents of admixtures combine with lime.

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[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

[2] Professor of Civil Engineering, In Charge of Engineering Materials Laboratory, University of California, Berkeley, Calif.

[3] Professor of Applied Mechanics, Kansas State College of Agriculture, Road Materials Laboratory, Manhattan, Kans.

[4] President, Colloy Products Co., St. Louis, Mo.

Certain admixtures containing high percentages of natural silica (90 per cent) have been shown to lack marked pozzuolanic properties, while substances lower in silica and higher in alumina have shown marked properties. It is only the total activated portion of the pozzuolana that is important. In general it is the percentage of non-crystalline (amorphous) materials, rather than the crystalline materials, which is important. The most effective blending medium is that in which the constituents silica, alumina, and ferrite are most highly activated, either naturally or artificially. Whether the silica be in the form of alpha, beta, or gamma quartz, or in a vitreous condition probably has some bearing on its activity.

The rational use of pozzuolana dictates that the optimum amount should be determined by the usual trial tests. The statement "No large reduction in cement content can be made, however, because workability, water-tightness and durability must be maintained at safe levels" deserves comment. Pozzuolanas, in considerable quantities per unit volume of concrete, are extensively used in major projects in foreign construction with considerably less cement per unit volume than is ordinarily used in this country for the same character of work. The durability of concrete made with pozzuolana is unquestioned abroad. It is well known that the monoforms of calcium silicate and aluminate such as formed by the union of free lime released during hydration of cement and pozzuolana are the most stable. Higher lime compounds (di- and tri- forms) are relatively unstable, being capable of further dissociation into free lime and mono-compounds. In addition, except when siliceous or aluminous blending mediums which detract from workability, or increase the mixing water requirements, are used, pozzuolanas will also from a purely physical standpoint improve water-tightness, reducing the percentage and sizes of voids. If the particles thereof are spheroidal, non-porous, and yet fine enough to have a large and active specific surface area the result is an increase in activity in combining with lime, with a resultant increase in water-tightness and chemical stability. Since the absolute volume of the average pozzuolana is greater than the absolute volume of an equal and optimum weight of the cement which it can replace, the absolute density of the resulting concrete in which it is used would be less than that of the concrete without the pozzuolanic blend or replacement, provided that not less than the normal amount of water were required for mixing. If the pozzuolana permits sufficient reduction in the water requirement for normal consistency or desired flow, the density is thereby increased and the percentage of voids (and also the void sizes) may be reduced. The important factor is that the soluble, reactive, free lime is combined to form insoluble, stable, cementing substances.

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[1] Shirchiro Nagai, "Studies of Mixed Portland Cement," *Journal*, Soc. Chem. Industry, Japan (Kōgyō Kwagaku Zasshi), Vol. 34, No. 5, May, 1931, p. 162-4B, Supplemental binding.

The general statement that "The use of higher percentages of silica, at least with the normal lime cement, requires the use of higher water-cement ratios," does not hold true in the case of certain pozzuolanic ash materials such as spheroidal fly ash, which is artificially activated by fusion at extremely high temperatures in the process of production. Tests on this pozzuolana indicate that slump and flow are increased as the percentage replacement of cement increases, provided the same volume of mixing water be used.

All pozzuolanas do not react with lime with equal rapidity or completeness. Their water factors vary greatly, as do also their fineness, grain-size distributions and specific surface areas, all of which greatly influence workability as well as both early and ultimate strength. The authors' strength results are not broadly applicable to all pozzuolanas—not even to the volcanic ash from various deposits.

Future tests on any one material might advantageously include higher curing temperatures since induration in many cases is complete in considerably less time than 28 days at these elevated temperatures. The elevation of temperature during curing increases the rate of hydration, that is, the decomposition of the high-lime constituents of the cement, and accordingly the percentage of lime released at early ages, making this available for reaction with any pozzuolana intimately available in the mix. The curing in a moist atmosphere at temperatures higher than those used in this investigation have, in the case of a spheroidal artificial ash, shown the following:

| | Series 11 | Series 12 | Series 13 | Series 14 |
|---|---|---|---|---|
| | No Replacement | 25 per cent Replacement | No Replacement | 6 per cent Addition |
| | Cured in molds 18 hr., thereafter in moist atmosphere at 150 to 160 F. (65 to 70 C.) for a period of 6 days only. | | Cured in molds 18 hr., thereafter in water at 70 F. (21 C.) for 7 days. | |
| Cement, g | 453 (100 per cent) | 340 (75 per cent) | 453 (100 per cent) | 453 (100 per cent) |
| Spheroidal ash, g | 0 | 113 (25 per cent) | 0 | 28 (6 per cent) |
| Sand, g | 1360 | 1360 | 1360 | 1360 |
| Water for given consistency[a] | 210 | 180 | 210 | 210 |
| Compressive strength, 2 by 4-in. cylinders, lb. per sq. in. | 3563<br>3627<br>3437 | 4518<br>4200<br>4200 | 2926<br>2738<br>2800 | 3054<br>3370<br>2926 |
| | 3542 | 4306 | 2820 | 3116 |
| | Increase, 21.5 per cent | | Increase, 10.5 per cent | |

[a] Consistency measured by Vicat method.

MESSRS. R. W. CARLSON[1] AND G. E. TROXELL[2] (*authors' closure, by letter*).—Mr. Bates makes a logical objection to the use of the expression "siliceous material" to designate volcanic ash, a particular form of silica. The writers have themselves noted that various siliceous materials have widely different properties which are not defined by chemical analysis alone. It is expected that the effect of form of silica will be investigated in future work.

In regard to the relative grindability of clinker of low-lime and high-lime content, comparisons have been made in which the minimum practicable burning temperatures have been used for the low-lime clinker. Even when burned at these lower temperatures, there still remains a large difference in grindability favoring the clinker of higher lime content. The authors know of no direct comparisons having been made to show the effect upon grindability of using less fluxing material for the low-lime clinker.

The authors agree with Mr. Hanna regarding the desirability of long-time tests of cements containing interground silica. It is believed that the effect of continued curing will be largely dependent upon the storage condition. Cores cut (after approximately 20 years) from a dam containing cement interground with granitic silica showed the interior concrete to have good strength with apparently no retrogression, while the concrete near the surface showed signs of disintegration. This is interpreted to mean that the particular blended cement used in this case was suitable under one condition of curing but was unsuitable for the severe exposure to which the outer concrete was subjected after only a brief period of curing.

In regard to Mr. Anderegg's suggestion that the percentage of active silica be determined, attempts are being made to find a test which will predict activity under the conditions which exist when the silica is present in hydrating cement. The tests customarily used to measure activity do not appear to do this.

Mr. Anderegg refers to Feret's conclusion that not more than 3 per cent of pozzuolanic material could be added in practice due to the extra water requirement. The writers have found this to apply only to certain materials, such as diatomaceous earths.

The writers did not intend to leave the impression that concrete made from portland cement is so pervious and so subject to disintegration as to have no enduring qualities. However, structures have been observed from which many tons of soluble materials have been leached by water under pressure percolating through the concrete.

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[2] Associate Professor of Civil Engineering, University of California, Berkeley, Calif.

While these are not examples of the best concrete, enough leaching action may be observed in nearly every concrete dam to place some value on the elimination of visible and invisible effects of soluble constituents in the cement.

The statement by Mr. Powers that the data of the report do not indicate the superiority of high-silica cement is concurred in by the authors. It might be added, however, that the data point to the possibility of producing blended cements which are superior to normal portland cements for certain classes of work. The data now available do not definitely establish this conclusion.

Mr. Powers is correct in stating that the effect of intergrinding volcanic ash with portland cement is to increase volume changes. The portland cement with which the blended cement should be compared, however, is not the one contained in the blended cement, but one which has properties like those of the blended cement: namely, a low-lime cement. As shown in this paper, a high-lime cement blended with volcanic ash exhibits appreciably less contraction than the low-lime plain cement.

It is not certain whether the slight retrogression in strength shown in Fig. 4 for one blended cement is real or is due to experimental error. Referring to Table IV, it may be seen that several cements, both plain and blended, show retrogression between the ages of three and six months. The one-year tests to be made in December, 1933, should prove whether or not these reductions in strength are significant.

The water content for these specimens was not held constant but was varied to give equal consistency as measured by a flow table. However, the differences in water content for the various cements tested were not great, as shown in Fig. 2. In general, the blended cements required slightly more water because of the greater bulk. In computing the water-cement ratios reported, the volcanic ash was considered as a part of the cement.

# THE DURABILITY OF CEMENT MORTARS; THE CEMENT AND METHOD OF TESTING MAJOR VARIABLES

By C. A. Hughes[1]

## Synopsis

The paper includes data on the absorptions, compressive strength, resistance to sodium sulfate and to three artificial cycles of freezing and thawing of mortars made from 18 brands of portland cement and 10 special cements. An attempt is made to show the relations, if any, existing between the above properties of the mortars. In addition, the relation of mortar durability as measured by immersion in 10 per cent sulfate or artificial freezing-and-thawing cycles to the calculated constitution and to Merriman's index of disintegration is discussed.

The demand for rapid hardening cement has led to a progressive modification of portland cement constitution and to the development of special high-early-strength cements. These changes consist chiefly of increases in tricalcic silicate and a consequent decrease in dicalcic silicate and have been accompanied by an increase in fineness.

The present investigation is an attempt to discover the relation between constitution and durability. The fact that there is no recognized laboratory standard for durability vastly increased the scope of the investigation. Previous work in this and other laboratories has indicated that relative durability depends to a considerable degree on the type of test and on the details of procedure. Consequently, it was decided to use a number of the better-known test procedures for durability. This resulted in making the method of test a variable of equal importance to the cement variable. While this investigation has been in progress for about two years, the data are not yet complete, nor have the data on hand been fully examined. Consequently, this paper must be regarded as a progress report.

### *Materials:*

*Cements.*—The cements included 18 portland and 10 special cements. Of these, 13 were bought on the Minneapolis market and 13 were contributed by various cement companies. Two cements were blends. Each sample of cement consisted of 3 sacks. They were thoroughly mixed as received and stored in covered cans in a dry room. In the report the cements are divided into two groups, portland and specials, and are numbered in order of increasing tricalcic silicate within each group, except Nos. 26, 27 and 28 for which the constitution could not be calculated.

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*Fine Aggregate.*—The fine aggregate was a local sand which has given satisfactory service over many years.

*Curing:*

Two types of curing were used for the mortars. Type 1 consisted of one day in the molds in the moist room, 13 days submerged in water, 11 days in air in the laboratory and 3 days in water. For this curing, all tests started at 28 days. In type 2 curing the specimens, after the initial one day in the molds in the moist room, were submerged in water until tested. For the strength specimens, the age at test was variable. All durability specimens of type 2 curing were given their first cycle or treatment at 90 days. All storage under water was done in a moist room, complying with specifications of this Society. For simplicity of reference, durability specimens given type 1 curing will be referred to hereafter as 28-day specimens, and those given type 2 curing as 90-day specimens.

*Weighing Specimens:*

Except for the dry and submerged weights on the absorption specimens, all weights were taken on saturated surface-dry specimens. The surface water and any loosened fragments on the durability specimens were blown off by use of an air hose operating under a pressure of 30 to 35 lb.

*Mortar Mixes:*

For durability tests, the water-cement ratio and consistency were considered to be of greater importance than the ratio of absolute volume of fine aggregate to absolute volume of cement, $\frac{a}{c}$. Therefore, it was decided to maintain constant water-cement ratios for all cements and to limit the variation in flow to 60 ± 10. This necessitated some variation in the $\frac{a}{c}$ ratios for the various cements.

Four water-cement ratios were used, 0.34, 0.42, 0.54 and 0.70 by weight. The mixes corresponding to these water-cement ratios will be referred to hereafter as the *B*, *C*, *D* and *E* mixes, respectively. The majority of durability tests were made on the *B*, *D* and *E* mixes.

All mixes were calculated from $\frac{a}{c}-\frac{w}{c}-$flow relations determined in advance. For the main group of mixes, the average flows were 44.8, 48.6, 55.1 and 55.6 and the standard deviations were 13.08, 9.92, 8.94 and 8.89 for the *B*, *C*, *D* and *E* mixes, respectively.

*Absorption Tests:*

Three cubes each of five mixes for six cements and three mixes for 22 cements were given the following treatment: At the completion of type 1 curing, they were weighed surface dry, placed in an oven at 105 to 110 C. for 24 hr., weighed, submerged for 48 hr., weighed surface dry, placed in boiling water for 5 hr., weighed submerged and again weighed surface dry. The changes in water content (weight) were calculated as ratios of the volume of the mortar. This permitted calculation of the absorptions by three commonly used methods, namely, saturated surface dry to dry, dry to saturated after 48 hr. immersion, dry to saturated after 5 hr. boiling. Hereafter the ratio of the volume of water absorbed from dry to 5 hr. boiling to the volume of mortar will be referred to as the porosity. The average values so obtained for the 18 portland cements were 0.171, 0.164, 0.165; 0.171, 0.166, 0.179; and 0.178, 0.172, and 0.179 for the *B*, *D* and *E* mixes, respectively. The individual values for the porosities are shown in Fig. 9.

For the portland cements, there is little difference between the values given by the three treatments for the 3 mixes of any given cement nor between the respective mixes of different cements. The differences are somewhat greater for the special cements. Since the $w_m$'s[1] for these specimens are nearly constant, it is not surprising that for portland cements which do not differ greatly in the extent of hydration at 28 days the absorptions should be nearly equal. For the special cements the difference between $w_m$ and the absorption is high and absorption correspondingly low when the strengths are above the average and *vice versa*. However, this approximation does not hold when some water-proofing compound is added as in the case of cement No. 23.

*Freezing-and-Thawing Tests:*

The freezing apparatus consisted of two 8-hole ice cream freezers. By operating the freezing units continuously the brine temperatures were maintained between 0 and −20 F. (−17.5 and −29 C.). Thawing was done in water at room temperature (70 to 75 F., 21 to 24 C.).

Three cycles of artificial freezing and thawing were used. Changes were made at or about 8:30 A. M., 12:30 P. M., and 5:30 P. M., so that freezing was done at the rate of 1½ cycles per day, long and short periods of freezing or thawing occurring alternately.

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[1] Volume of mixing water expressed as a ratio of the volume of mortar.

The *H* cycle consisted of a period submerged in water at room temperature and under pressure of 150 lb. per sq. in., followed by a period in air at 0 F. (−17.5 C.) ± 10 F. (±5.5 C.) in the freezer. The pressure is used to increase the water content of the specimens at the time of freezing, and hence to increase the rate of disintegration. This cycle and the effect of pressure has been discussed more fully elsewhere.[1]

The *S* cycle was similar to the *H* cycle except that the cubes were stored in water at atmospheric pressure during the thawing period. This cycle closely approaches natural freezing and thawing on saturated concrete as at the water-line or ground line of structures. The only objection to it is the large number of cycles necessary to obtain a relative measure of resistance to freezing and thawing.

In the *F* cycle, the cubes were submerged in water in metal cans. The cans were placed alternately in a freezing chamber containing alcohol solution at −10 F. (−23 C.) ± 10 F. (±5.5 C.) and in water at room temperature. This cycle is similar to that used by Scholer[2] and to the Society's proposed Method of Testing Concrete and Concrete Aggregates by Freezing and Thawing.[3] A serious practical objection to this method is the heavy loss of containers due to bulging and consequent leaking. It is recognized that the deep cans used in this investigation exaggerated this condition.

Since it was known from readings of thermocouples placed at their centers that cubes froze more rapidly at the bottom of the rack or can than at other positions, the cubes of the *H*, *S* and *F* cycles were shifted in position once per week or about every 10 cycles, those from the bottom being moved to the top and the remainder one shelf downward. Since the thermocouple readings had shown that there was no practical difference in temepratures at top and middle positions for the *S* and *H* cycles and little differences for the *F* cycle, it is believed that this procedure eliminated any significant effect due to temperature differences within a given cycle.

*Natural Weathering:*

The natural weathering cubes were placed outside the laboratory in a wire enclosure. Two cubes of each mix were placed in $\frac{3}{4}$ in. of water in order to obtain a water-line condition since damage to concrete due to freezing and thawing has not been observed by the author except where the concrete or mortar is saturated. Two more cubes of each mix were placed in air.

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[1] F. C. Lang, and C. A. Hughes, "Discussion on Relation Between Durability of Concrete and Durability of Aggregates," *Proceedings*, Tenth Annual Meeting, Highway Research Board, p. 116 (1930).

[2] C. H. Scholer, "The Durability of Concrete," *Proceedings*, Tenth Annual Meeting, Highway Research Board, p. 146 (1930).

[3] C. H. Scholer and A. E. Stoddard, "Proposed Method of Testing Concrete and Concrete Aggregates by Freezing and Thawing," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 364 (1932).

*Expansion of Beams:*

A limited number of 2 by 2 by 10-in. mortar beams were included in the *H* cycle, *S* cycle and natural weathering series. These were fitted with brass plugs at each end and the changes in length measured by a simple apparatus consisting essentially of a 0.0001-in. dial on a suitable mounting. In order to eliminate temperature effects, all measurements were made at the end of the thawing period and with the specimen and instrument submerged in water at 70 F. (21 C.)

*Sodium Sulfate Tests:*

A full series of cubes were placed in a 10 per cent sodium sulfate solution. This test has been used[1] as a measure of durability, and it was desired to find what relation, if any, existed between resistance to sodium sulfate and resistance to freezing and thawing. The cubes were rated visually and weighed once per month.

In the visual inspection four stages were recognized as follows: Rating 1, single-line crack on one edge; rating 2, single-line crack on 4 edges; rating 3, multiple-line cracks or warping; rating 4, one or more rounded edges. Since one-half ratings were recorded there were 8 possible assignable values. Visual inspection was discontinued after a rating of 4 had been given.

At the time ratings were made considerable doubt was felt as to their value, since the method of disintegration varied with different cements. The majority followed the method of rating, but others merely softened at the edges, while others checked and expanded in volume. It was believed, and still is, that the number of days immersion for a given loss in weight gives a more reliable index of resistance than does any method of visual rating. While weights do not always reflect the condition of the specimen (see Fig. 3, cement No. 3) at least they will give consistent results between different operators, a condition which is most unlikely for any method of visual inspection. Despite these objections, the measures of resistance obtained by weights and by inspection for a limited number of specimens were in agreement to a surprising degree.

*Merriman's Index of Disintegration:*

The methods used in this test conformed with those outlined in papers by Merriman[1] and Carlson and Bates.[2] The tests were made, under the author's direction, by a graduate in chemistry holding a master's degree. Considerable difficulty was experienced in getting checks between duplicate tests until the certain details of the procedure, particularly the time element, were rigidly standardized. That success was finally achieved is shown by the correlation coefficients[1] of 0.986, 0.987, 0.980 for the first and second determinations of Merriman's index for the 18 portland cements, 9 special cements, and the 27 cements of both groups, respectively.

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[1] Thaddeus Merriman, "Durability of Portland Cement," *Engineering News-Record*, January 9, 1930, p. 62.

[2] E. T. Carlson and P. H. Bates, "Can Cement Durability Be Predicted," *Engineering News-Record*, July 23, 1931, p. 130.

*Measures of Resistance to Freezing and Thawing and to Sodium Sulfate Resistance:*

When materials differing widely in resistance to a given treatment are used, the only practical measure of relative resistance is the number of cycles or number of days immersion required to cause a given degree of disintegration. The choice of the degree of disintegration presents a problem. In freezing-and-thawing cycles in which all specimens increase in weight, as in the *H* cycle, the number of cycles at maximum weight or at zero per cent change in weight may be used. These measures have the advantage of giving results with a minimum number of cycles. However, they depend too largely on small differences in weight and are not believed to give so consistent results as the number of cycles at an appreciable loss in weight.

In this investigation the number of cycles at 30 per cent loss in weight is used as the measure of resistance for the *H* and *F* cycle cubes and 5 per cent for the *S* cycle cubes since this cycle is not yet advanced far enough to permit the use of 30 per cent. For the beams the number of cycles at an expansion of 0.01 in. was adopted after comparing the results at 0.002,and 0.03 in. An expansion of 0.01 in. is 1000 points on the dial as read and is a relative expansion of 0.001 since the beam length is 10 in. The number of cycles at zero per cent loss in weight was obtained for the *H* cycle in order to show something of the effect of changing the degree of disintegration.

For the sodium sulfate cubes the number of days at rating 3 was used since this series is not complete enough to permit the use of a weight measure. The rating used for each mix is the average of ratings on the three specimens of each group.

In certain comparisons the "average" of the number of cycles or days immersion for the *B*, *D* and *E* mixes has been used as an overall index of the resistance of the cement to the given treatment.

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[1] Report of Committee C-8, Appendix: Manual for Interpretation of Refractory Test Data (1929), *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 478 (1930).

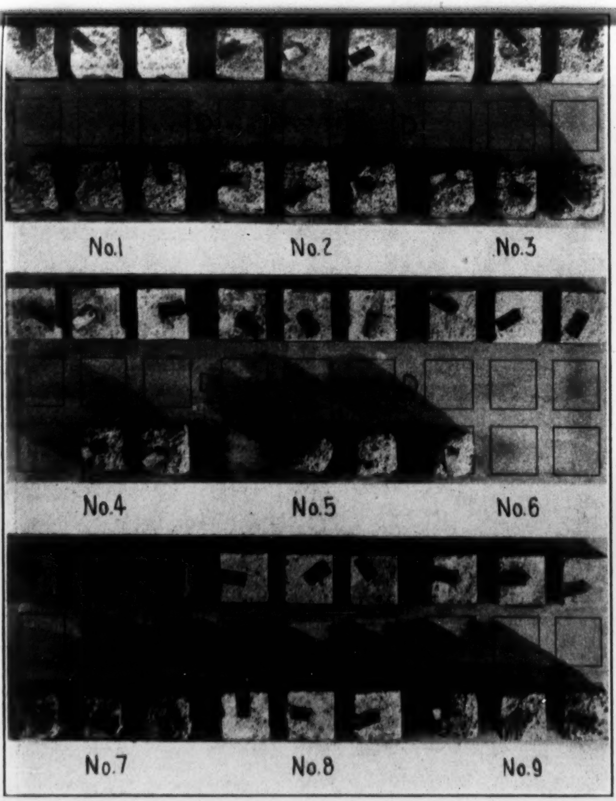


FIG. 1.—Typical Appearance of the 90-day *S* Cycle Cubes After the Number of Cycles of Freezing-and-Thawing Given Below.

**In each group, the *B* mix specimens are in the top row and the *E* mix specimens are in the bottom row. No *D* mix specimens were used in this cycle. The blank spaces in the bottom row indicate specimens that have completely disintegrated.**

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Cement Number... | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| Number of Cycles.. | 193 | 215 | 212 | 241 | 220 | 186 | 243 | 182 | 203 |

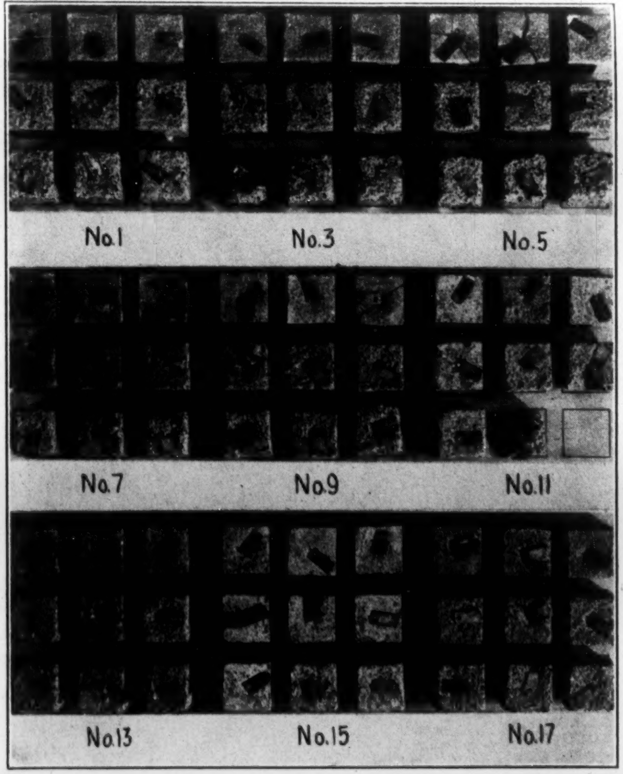
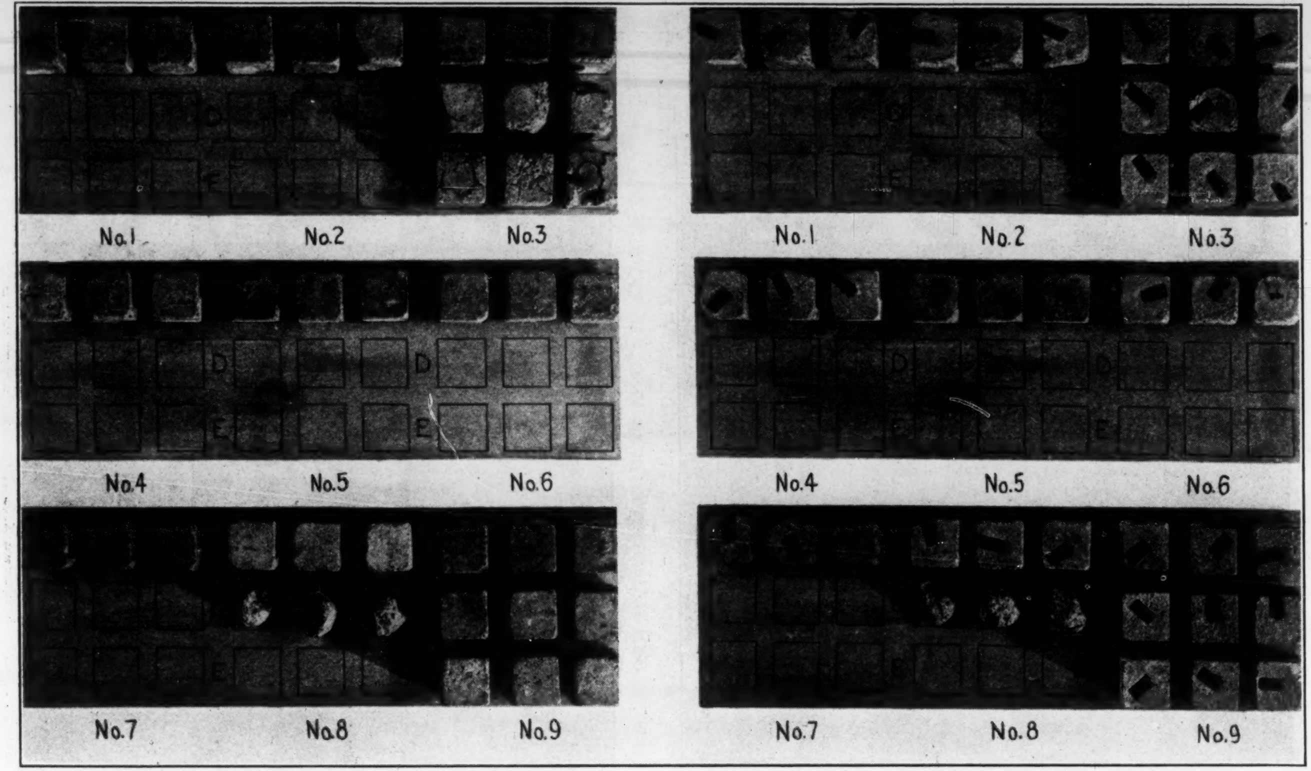


FIG. 2.—Typical Appearance of the 90-day *F* Cycle Cubes After the Number of Cycles of Freezing-and-Thawing Given Below.

**In each group the *B* mix specimens are in the top row, the *D* mix specimens in the center row and the *E* mix specimens are in the bottom row. Note the splitting of the specimens.**

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Cement Number... | 1 | 3 | 5 | 7 | 9 | 11 | 13 | 15 | 17 |
| Number of Cycles.. | 23 | 30 | 32 | 26 | 30 | 26 | 26 | 17 | 23 |


No.1
No.2
No.3
No.4
No.5
No.6
No.7
No.8
No.9
D
E
No.1
No.2
No.3
No.4
No.5
No.6
No.7
No.8
No.9
D
E

(*a*) Bottoms of cubes. (*b*) Tops of cubes.

FIG. 3.—Typical Appearance of the Cubes After Immersion in 10 per cent Sodium Sulfate Solution.

In each group the *B* mix specimens are in the top row, the *D* mix specimens in the center row and the *E* mix specimens are in the bottom row. The blank spaces indicate cubes that were entirely disintegrated.

| Cement Number... | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| Days............. | 209 | 227 | 224 | 246 | 231 | 204 | 248 | 201 | 217 |

*Types of Disintegration:*

The appearance of some of the specimens during the course of the tests is shown in Figs. 1, 2 and 3. The appearance of the *H*-cycle specimens is similar to those for the *S* cycle except that the scaling is more pronounced.

In the freezing-and-thawing tests there appeared to be three general types of disintegration which may be described as follows:

*Type 1.*—Progressive scaling from the outside leaving a hard core impervious to air. This is the type typical of the *H* cycle specimens at 28 days and for the intermediate and lower water-cement ratios. Weight decreases slowly over a lengthy period.

*Type 2.*—Marked checking throughout the body of the specimen. Air can often be blown through specimens before there is any loss in weight. Weight decreases are sudden and marked. In some cases specimens show increase in weight at one weighing and disintegrate completely at the next weighing. Material is removed in irregular fragments of considerable size ($\frac{1}{8}$ to $\frac{1}{2}$ in. per side) which tend to be cubical. This is typical of 90-day *H* specimens, particularly those of the lower water-cement ratios and the 28-day low water-cement ratio mortars.

*Type 3.*—This type of disintegration is peculiar to the *F* cycle. In the *B* mixes there is pronounced splitting with little or no loss in weight. In the *E* mixes there is some splitting always accompanied by considerable loss in weight. The paste is removed leaving aggregate particles protruding which are easily rubbed off. The core tends to be soft but is impervious to air. A stage is reached at which specimens still of considerable size can be easily crumbled by the hand. The behavior of the *D* mixes is intermediate between those of the *B* and *E* mixes. This disintegration, particularly the splitting, is not thought to be similar to the disintegration resulting from natural weathering.

The appearance of the cubes of the *F* cycle is very similar to that of cubes frozen by direct immersion in calcium chloride brine. The difference in the disintegration between this cycle and the cubes of the *H* and *S* cycles is believed to be due to the very rapid rate of freezing. From thermocouple readings, it was determined that the center of the specimens, other than those at the bottom of the freezing chamber, reach 24 F. (−4.5 C.) in about 185 minutes for the *S* and *H* cycle cubes and in about 16 minutes for the *F* cycle cubes. The corresponding times for the bottom cubes is 84 minutes and 9 minutes, respectively.

## TEST DATA

The average data from the $H$ and $F$ cycles for the 18 portland cements are shown plotted against the $\frac{c}{v + c}$ ratio in Fig. 4. Before using the $\frac{c}{v + c}$ ratio the relation with the water-cement ratio was examined and found to be less suitable than that with the $\frac{c}{v + c}$ ratio. The relationship is of the form:

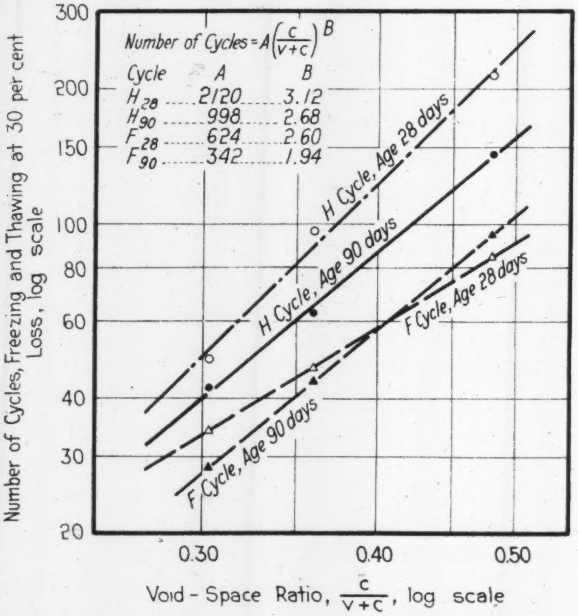


FIG. 4.—Relations Between the Resistance to Freezing and Thawing by the $H$ and $F$ Cycles and the $\frac{c}{v + c}$ Ratio.

The graphs and equations are for the averages of the data for the 18 portland cements.

$$N = A\left(\frac{c}{v + c}\right)^B$$

in which $N$ is the number of cycles and $A$ and $B$ are constants depending on the cement and the method of testing. Values of $A$ and $B$ for the average curves of the 18 portland cements are given in the figure. In this and similar cases the curves were fitted by least squares.

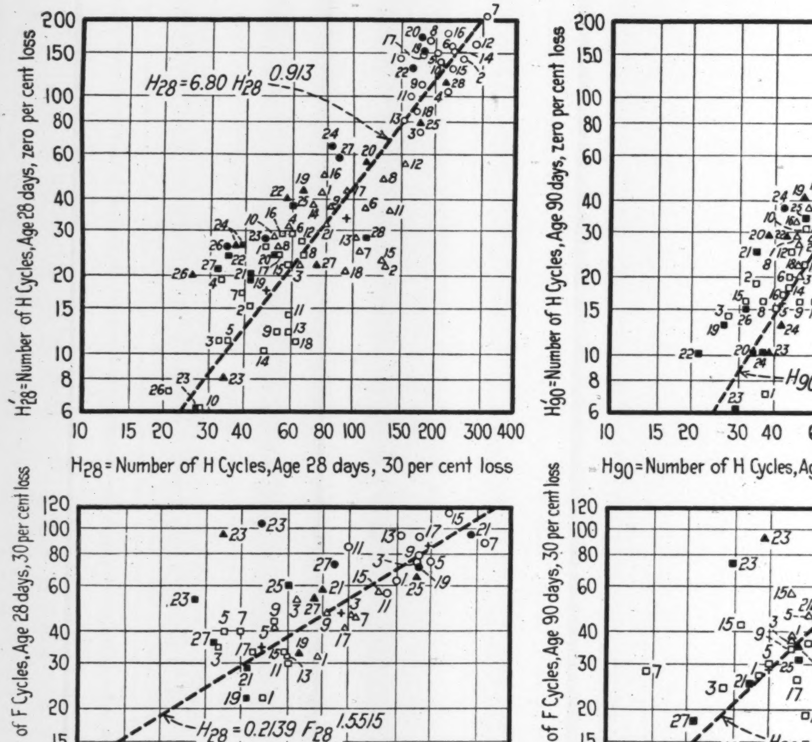
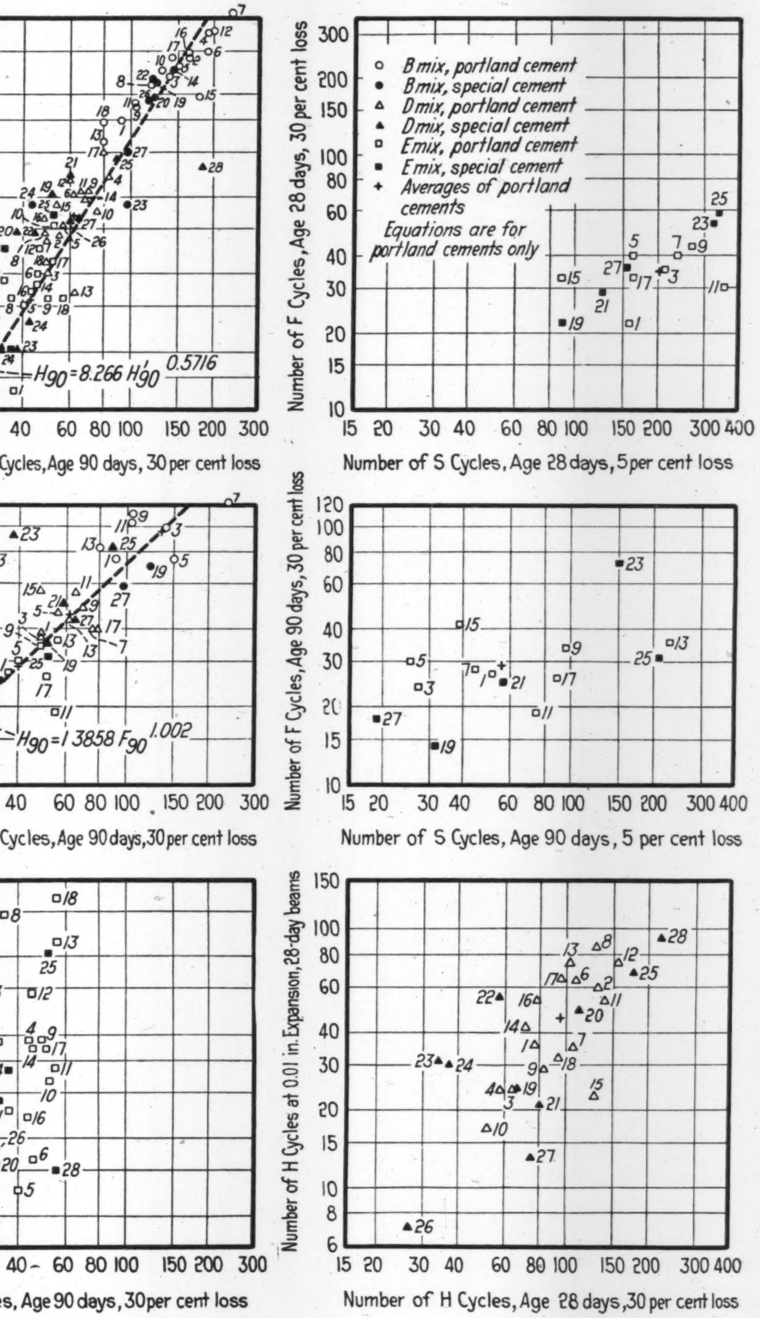


FIG. 5.—Relations Between Various Cycles and Methods of Measuring Resistanc

The dashed lines and equations are for the averages for the 18 portland cements of each

**PLATE I.**
**PROCEEDINGS, AM. SOC. TESTING MATS.**
**VOL. 33, PART II**
**HUGHES ON DURABILITY OF CEMENT MORTARS**



esistance to Freezing and Thawing. The curves are all plotted to log scales.

f each group of mixes.

The surprising fact shown by Fig. 4 is that the resistance of the 90-day specimens is in general less than for the 28-day specimens. The author fully expected the reverse. Since considerable leaching of the specimens was observed in the moist room, this was examined as a possible explanation of the greater resistance of the 28-day specimens. However, little consideration was necessary to realize that leaching would be as great during the freezing-and-thawing cycles as during storage.

The author was fortunate in having a few data from another investigation which suggested an explanation. These data are given in Table I. The data of Table I show the increase in resistance to be expected from longer curing. They show also that 10 days in air storage increases the resistance to freezing and thawing more than 26 days additional moist curing. Using the argument that shrinkage on drying would cause stress, if not cracks, in the paste between the aggregate particles, it was expected that the 10-day air curing would reduce the resistance to freezing and thawing. Instead the reverse occurred.

TABLE I.—THE EFFECT OF AIR STORAGE ON THE RESISTANCE OF MORTARS TO FREEZING AND THAWING BY THE *H* CYCLE.

| Curing | | Number of *H* Cycles at 30 per cent Loss | | | |
|---|---|---|---|---|---|
| Submerged in Water, days | In Air, days | Cement A | | Cement B | |
| | | Water-Cement Ratio by Weight =0.665[a] | Water-Cement Ratio by Weight =0.887[b] | Water-Cement Ratio by Weight =0.665[a] | Water-Cement Ratio by Weight =0.887[b] |
| 2 | 0 | 10 | .. | 24 | .. |
| 2 | 10 | 24 | .. | 63 | .. |
| 28 | 0 | 19 | 11 | 53 | 21 |
| 28 | 10 | 38 | 22 | 67 | 39 |

[a] Average of 6 groups of 3 specimens each.
[b] Average of 3 groups of 3 specimens each.

It appears, therefore, that the increased resistance of the 28-day specimens is due to the 14 days of air curing included in the curing of the 28-day specimens. Increased resistance due to air curing, has been observed also in sulfate tests.[1]

The effect of a period of air curing varied somewhat with the various cycles and measures of resistance. It affected but little the average *H* cycle resistance at zero per cent loss. The greatest effect on the average *H* cycle mixes at 30 per cent loss occurred for the richest mix; the reverse was true for the *F* cycle at 30 per cent loss. In this case the richer mixes had greater resistance on the average at 90 days than at 28 days. The *E* mixes of the *S* cycle showed the greatest difference of the three cycles. The boiling and oven drying given the absorption specimens markedly reduced the resistance of the *D* specimens to the *H* cycle whereas only the lower-resistant specimens of the *E* mix showed decreased resistance to the *F* cycle.

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[1] D. G. Miller and P. W. Manson, "Laboratory and Field Tests of Concrete Exposed to the Action of Sulphate Waters," *Technical Bulletin No. 357*, U. S. Department of Agriculture.

Evidently the argument intended to apply to air storage is valid for oven drying. That shrinkage cracks had developed in the absorption specimens was obvious during testing since in many specimens air could be blown through them.

Some relations resulting from different methods of testing and test procedure are shown in Fig. 5 (Plate I). The dashed lines and equations apply to the data for the portland cements only. It will be observed that they give the relation between the average for each of the three mixes. Closer examination shows that relations exist between the three mixes of each individual cement similar to that for the averages, but, of course, with different constants. Among the mortars of the same water-cement ratio made from different cements similar relations exist, but again with different constants. In general, these constant water-cement ratio group relations are rather vague for this figure, but some are clearly defined as for example the *B* mixes of the top center diagram and the *E* mixes of the top right diagram.

Since the relations between the various cycles or procedure vary with the brand of cement, the correlations of the various cycles can be shown only by the data from mixes of the same cement. However, there is sufficient evidence to show that there is some degree of correlation between the various cycles and procedures.

That extreme caution should be exercised in making comparisons between various materials from data on freezing-and-thawing tests until more exact information is obtained than is now available is well illustrated by the reference to the data on the *D* mix of cement No. 2 shown in the upper left diagram of Fig. 5. The resistance of this cement is nearly the highest for this mix at 30 per cent loss whereas it is nearly the lowest at zero per cent loss.

During freezing and thawing, conditions favorable for continued curing exist for better than 50 per cent of the period. It follows that the true relation between strength and resistance to freezing and thawing cannot be obtained from the use of the strength at the beginning of test.

Figure 6 shows the relation between the compressive strength of

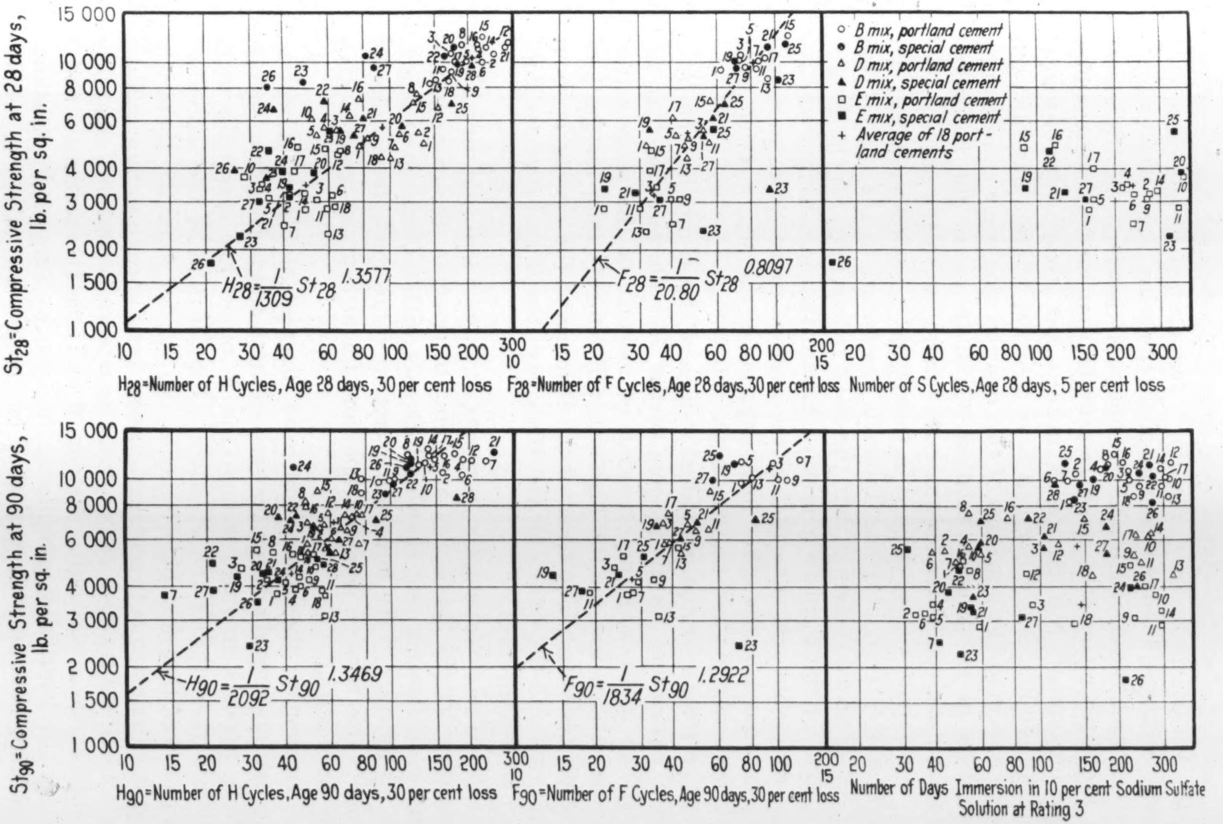

FIG. 6.—Relation Between the Compressive Strength at the Beginning of Test and the Resistance to Freezing and Thawing and to Immersion in 10 per cent Sodium Sulfate Solution.

the cubes at the beginning of the testing period and the resistances to the various tests for durability. The dashed lines indicate the average relation between compressive strength at the beginning of the test and the resistance to freezing and thawing. If the points for the three mixes of a given cement be examined, as for example those for cements Nos. 26 and 23, in the upper left diagram of Fig. 6, they will be found in general to have a similar relation but values of the constants in the equation of this relation may vary considerably from that of the average relation. The trends of the individual values for a given water-cement ratio referred to in the discussion of Fig. 5 are quite clearly defined in this figure. Taking the upper left diagram the trend of the *E* mixes is downward to the right; that for the *D* mixes is similar, but the slope is less than for the *E* mix; and that for the *B* mixes is upward toward the right with a positive slope somewhat less than that of the average curve. Similar trends may be traced in the other four diagrams of this figure which show data from freezing-and-thawing tests.

The trends for the individual values of the *E* mix for the *H*, *S* and *F* cycles and for the *D* mix of the *H* cycle indicate that the resistance to freezing and thawing of mortars of the same water-cement ratio is less for the high-strength cements than for the low-strength cements. The trend of the *D* mixes of the *F* cycle is about horizontal and the trends for the *B* mixes of the *H* and *F* cycles slope more or less upward.

Briefly summarizing the foregoing, it may be said that for a given cement or for the average of a number of cements, the resistance of mortars to freezing and thawing increases with increase in strength, but for mortars of the same water-cement ratios made from different cements this relation does not hold and for high water-cement ratios may be reversed.

Turning to the lower right-hand diagram of Fig. 6, it will be seen that no general relation between strength and resistance to sodium sulfate appears to exist though the *B* mixes are markedly more resistant than the *D* and *E* mixes. Examination of the individual points show that for some cements, No. 12 for example, there is a very good relation between strength and resistance to sodium sulfate, whereas for others such as No. 9 the resistance is constant regardless of the change in strength. Reference to Table III, sections 2 and 3 reveals that, with few exceptions, the cements varying in resistance to sodium sulfate with variation in compressive strength (water-cement ratio) are those of low resistance to sodium sulfate and those whose resistance is independent of strength are those of high resistance to sodium sulfate.

The diagrams of Fig. 7 show the relations between resistance to sodium sulfate and resistance to freezing and thawing. In these diagrams, the averages of the *B*, *D* and *E* mixes have been used except for the *S* cycle, for which data on the *E* mix only are available.

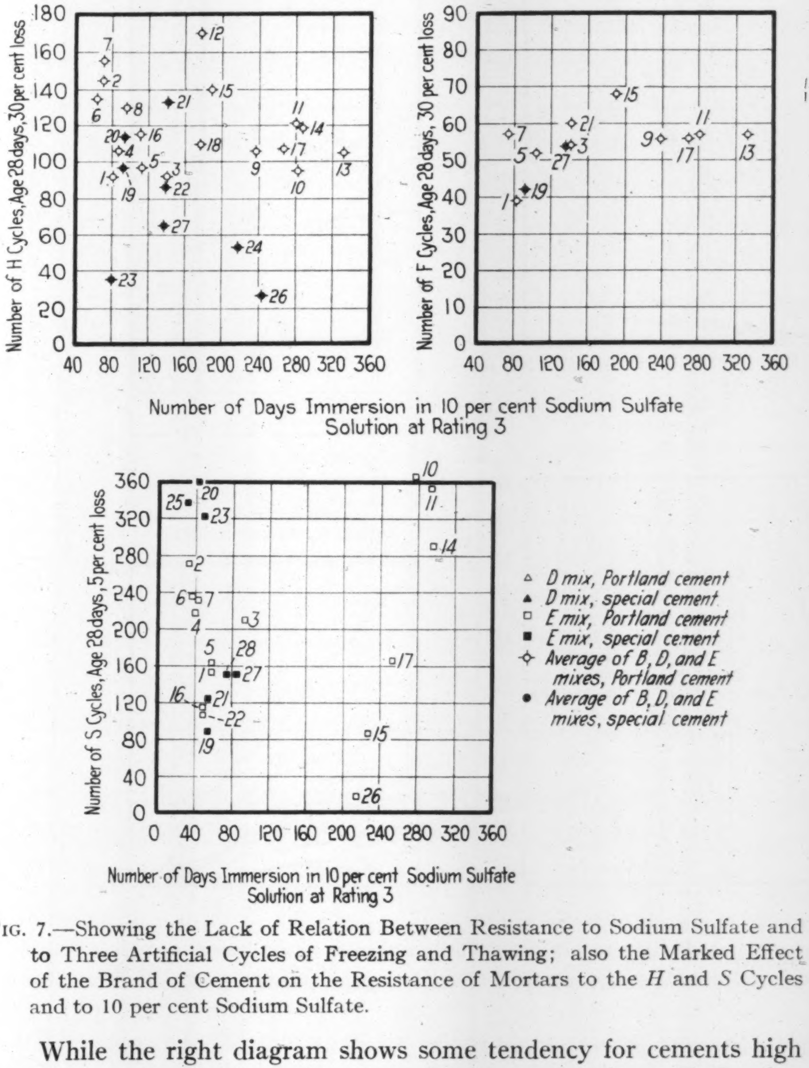

FIG. 7.—Showing the Lack of Relation Between Resistance to Sodium Sulfate and to Three Artificial Cycles of Freezing and Thawing; also the Marked Effect of the Brand of Cement on the Resistance of Mortars to the *H* and *S* Cycles and to 10 per cent Sodium Sulfate.

While the right diagram shows some tendency for cements high in sulfate resistance to be low in resistance to freezing and thawing by the *H* cycle, there is evidently little, if any, relation between the resistance to sodium sulfate and the resistance to freezing and thawing.

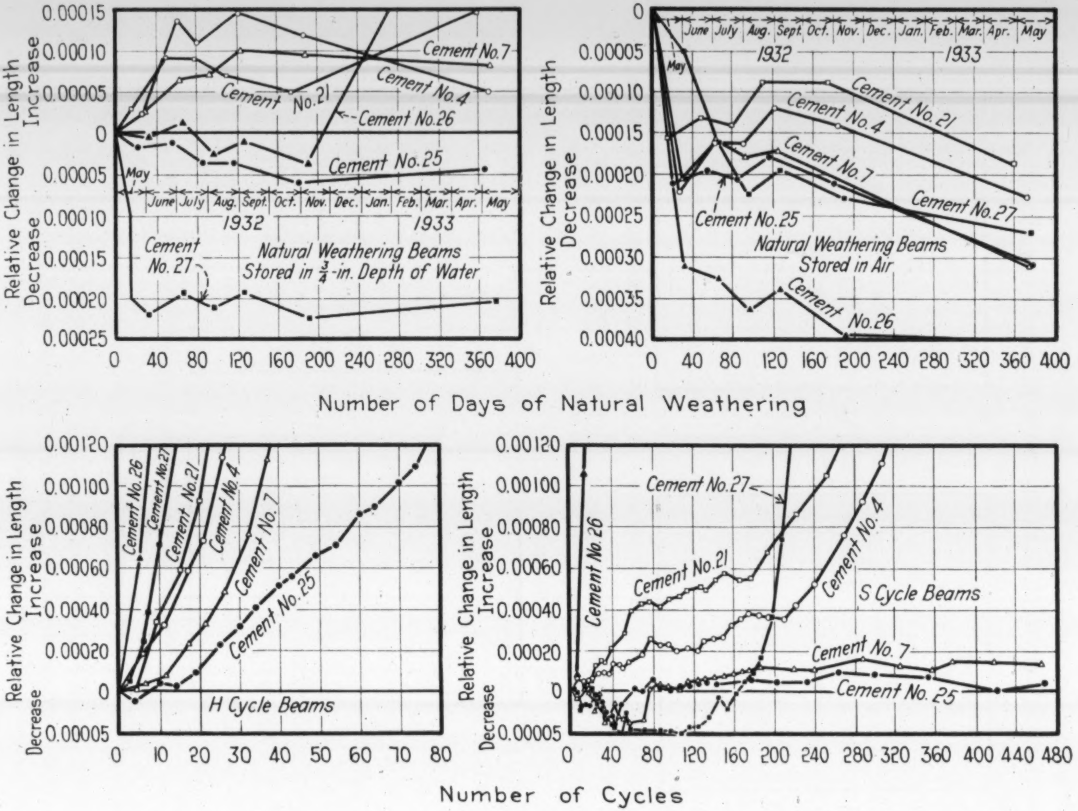

FIG. 8.—Relative Linear Changes in 2 by 2 by 10-in. Mortar Beams Due to Freezing and Thawing and to Exposure to Natural Weathering.

Each point represents the average of readings on two beams. Note that cement No. 26 which has the least resistance to the two artificial cycles shows marked expansion due to outside exposure for one winter.

The diagrams of Fig. 7 show clearly the marked influence of the brand of cement on the resistance to freezing and thawing. It will be noted from the figure that the effect of the brand of cement varies with the type of cycle used. In the *F* cycle, nine of thirteen cements lie between 50 and 60 cycles. The spread of resistances in the *H* and *S* cycles is much greater. It is therefore apparent that until an artificial cycle is definitely correlated with natural weathering, conclusions drawn from the data of laboratory tests should not be taken too seriously.

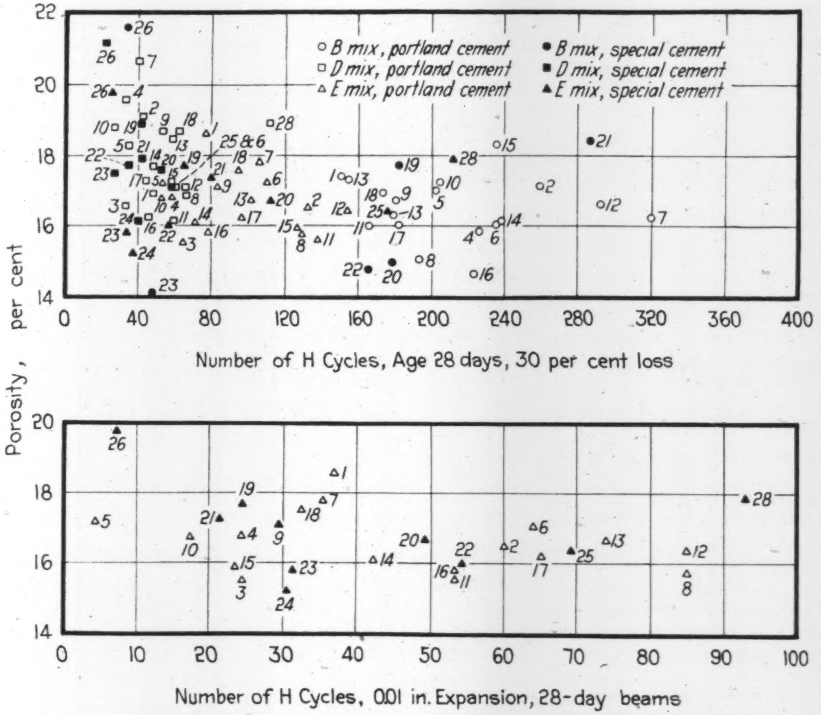

FIG. 9.—Porosity *versus* Resistance to Artificial Freezing and Thawing.

The natural weathering specimens have not been exposed long enough to yield any definite information. The graphs of the natural weathering beams are shown in the two upper diagrams of Fig. 8. It will be seen that those in air have shown no expansion, but are still shrinking. Of those partly submerged, only Nos. 21 and 26 are showing any appreciable expansion and of these the expansion of No. 26 is by far the greater. Cement No. 27 has shown considerable shrinkage. The behavior of cements Nos. 21, 26 and 27 in the natural weathering, *H* cycle and *S* cycle are so far closely similar and lead to the hope that correlation between natural weathering and the artificial cycles will be established. In an attempt to obtain some idea of the relation between the number of artificial cycles and the duration of natural weathering, the number of *H* and *S* cycles resulting in expansion equal to those given by natural weathering over one winter were determined. It was found that the number of cycles so determined for cement No. 21 were 6 and 25 and for cement No. 27, 4 and 7 for the *H* and *S* cycles, respectively.

TABLE II.—COEFFICIENTS OF THE FOUR MAJOR CONSTITUENTS OBTAINED BY LEAST SQUARE SOLUTIONS FROM DATA ON 18 PORTLAND CEMENTS, EXCEPT AS NOTED.

$C_3S = 3CaO \cdot SiO_2$ $C_3A = 3CaO \cdot Al_2O_3$
$C_2S = 2CaO \cdot SiO_2$ $C_4AF = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$

| Experimental Quantity | Coefficient of $C_3S$ | Ratios of Coefficients to Coefficient of $C_3S$ | | | | Residuals of Experimental Values, per cent | | |
|---|---|---|---|---|---|---|---|---|
| | | $C_3S$ | $C_2S$ | $C_3A$ | $C_4AF$ | Mean | Range | |
| | | | | | | | Plus | Minus |
| Number of *H* cycles at 30 per cent loss, age 28 days—average of *B*, *D* and *E* mixes | +2.719 | +1 | +0.41 | +1.14 | −2.73 | 12 | 20 | 28 |
| Number of *H* cycles [illegible] per cent loss, age 90 days—average of *B*, *D* and *E* mixes | +1.623 | +1 | +0.56 | +1.19 | −2.45 | 13 | 39 | 17 |
| Number of *S* cycles at 5 per cent loss,[a] age 28 days—*E* mix | +8.499 | +1 | +1.23 | −1.43 | −4.21 | 27 | 112 | 36 |
| Number of *S* cycles at 5 per cent loss,[a] age 90 days—*E* mix | +3.614 | +1 | +1.03 | −1.71 | −4.28 | 20 | 70 | 24 |
| Number of *F* cycles at 30 per cent loss,[b] age 28 days—average of *D* and *E* mixes | +1.288 | +1 | −0.08 | +0.31 | −0.33 | 6 | 16 | 13 |
| Number of *F* cycles at 30 per cent loss,[b] age 90 days—average of *D* and *E* mixes | +0.495 | +1 | −0.20 | +1.23 | +2.42 | 9 | 26 | 20 |
| Number of *H* cycles −0.01 in. expansion, age 28 days—*D* mix beams | +1.318 | +1 | +0.30 | +0.43 | −2.63 | 66 | 374 | 45 |
| Number of days rating of 3 $Na_2SO_4$ 10 per cent solution—average of *B*, *D* and *E* mixes | +5.559 | +1 | +0.40 | −4.13 | +1.87 | 28 | 85 | 33 |
| Merriman's index | +0.034 | +1 | −3.20 | +18.09 | +9.36 | 14 | 18 | 100 |

[a] Fourteen portland cements.
[b] Nine portland cements.

Plotting the data from the freezing-and-thawing tests and the sodium sulfate tests against Merriman's index failed to show any significant relation, though there appears to be some tendency for the resistance to sodium sulfate to be low and the resistance to the *H* cycle to be high for high values of the index.

The porosities or absorptions by volume after 5 hr. boiling are plotted in Fig. 9 against the data from two freezing-and-thawing cycles. Since the variation in porosity is small, no direct data are given on the effect of porosity on the resistance to freezing and thawing. However, it is very clearly demonstrated by Fig. 9 that for mortars of about the same consistency wide variations in resistance to freezing and thawing may be experienced within a narrow range of porosity. The above data would lead to serious doubt as to the use of the absorption test as a measure of the resistance of mortars to freezing and thawing.

## DISCUSSION OF RESULTS

### *Relation Between Composition, Constitution and Resistance to Freezing and Thawing and Resistance to Sodium Sulfate:*

In an attempt to relate the resistance to freezing and thawing, least square solutions were made to obtain equations relating the various resistances of the 18 portland cements to the four major constituents. Data from these solutions are given in Table II. Since these equations are regarded as chiefly useful in indicating the relative effect of the four constituents, the coefficients are expressed as ratios of the coefficient of $C_3S$[1] in order to permit easier comparison. The coefficient of $C_3S$ is also given so that the actual coefficients may be readily obtained if desired. In order to give some idea of the magnitude of the residuals (the differences between the values calculated from the equations and the experimental values) the means and ranges expressed as percentages of the experimental values are shown.

Another attempt to find a relation between the constitution and the resistance to freezing and thawing or to sodium sulfate was made by arranging the data in various groups and averaging the characteristics of each group. These data are given in Table III.

### *Relation of Sodium Sulfate Resistance to Constitution:*

It will be noted in Table II that the coefficients from the least square solution for the average resistances of the *B*, *D* and *E* mixes to 10 per cent sodium sulfate solution are positive, except that for $C_3A$.[2] The coefficients for $C_3A$ and $C_4AF$[3] are also greater than for $C_3S$ and $C_2S$.[4] The coefficients for the equations of the individual mixes were essentially similar to those for the average resistances of the three mixes.

The coefficients of the least square solution suggested that a relation might be obtained using either tricalcium aluminate alone

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[1] $C_3S = 3CaO \cdot SiO_2$.

[2] $C_3A = 3CaO \cdot Al_2O_3$.

[3] $C_4AF = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$.

[4] $C_2S = 2CaO \cdot SiO_2$.

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TABLE III.—RELATIONS BETWEEN THE AVERAGE CHARACTERISTICS OF CEMENTS FOR CONDITIONS INDICATED IN SUBTITLES.

Significant Values are Given in Bold-Faced Type.

| Group | Cement Numbers | $Na_2SO_4$ Average Number of Rating 3, Average of *B*, *D* and *E* Mixes | Number of Freezing-and-Thawing Cycles |  |  |  |  |  |  | $C_3S$, per cent | $C_2S$, per cent | $C_3A$, per cent | $C_4AF$, per cent | $CaSO_4$, per cent | Uncombined Lime, per cent | Silicates, per cent | $R_2O_3$, per cent | $\frac{SiO_2}{R_2O_3}$ | $\frac{Al_2O_3}{Fe_2O_3}$ | $\frac{CaO}{SiO_2+R_2O_3}$ | Molecular Ratio | Loss on Ignition, per cent |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  | Cubes, *H* Cycles, 30 per cent Loss, Average of *B*, *D* and *E* Mixes |  | Cubes, *S* Cycle, 5 per cent Loss, *E* Mix |  | Cubes, *F* Cycles, 30 per cent Loss, Average of *B*, *D* and *E* Mixes |  | Beams, *H* Cycle, Expansion 0.01 in., *D* Mix |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  | 28 days | 90 days | 28 days | 90 days | 28 days | 90 days | 28 days |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 1. CEMENTS IN LOWER AND UPPER GRAPHS OF FIG. 10 (*b*). |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Upper Graph.. | 5,9,12,13,14,16 | 209.8 | 119 | 82 | 210 | 105 | 55 | 56 | 48 | 46.7 | 25.5 | 10.4 | 9.20 | 3.20 | 1.16 | 72.1 | 8.89 | 2.41 | 1.91 | 2.13 | 2.65 | 1.38 |
| Lower graph.. | 1, 2, 3, 4, 6, 7, 8, 10, 11, 15, 17, 18...... | 150.1 | 119.1 | 83 | 220 | 104 | 55 | 55 | 43 | 45.0 | 27.8 | 10.2 | 8.85 | 3.02 | 1.03 | 72.7 | 8.64 | 2.51 | 2.05 | 2.12 | 2.63 | 1.61 |
| 2. IMMERSION IN 10 PER CENT SOLUTION OF SODIUM SULFATE. AVERAGES OF *B*, *D*, AND *E* MIXES. GROUP No. 1—RESISTANCE VARIES WITH WATER-CEMENT RATIO. GROUP No. 2—RESISTANCE INDEPENDENT OF WATER-CEMENT RATIO. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Group No. 1.. | 1,2,3,4,5,6,7,8, 12, 16...... | [a] | 124 | 86 | 201 | 86 | 50 | 53 | 47 | 42.8 | 28.9 | **11.97** | 8.38 | 3.08 | 0.94 | 71.7 | 9.04 | **2.39** | **2.30** | 2.10 | 2.63 | 1.13 |
| Group No. 2.. | 9, 10, 11, 13, 14, 15, 17...... | [b] | 113 | 80 | 254 | 97 | 59 | 59 | 43 | 48.1 | 25.6 | **8.00** | 9.70 | 3.00 | 1.27 | 73.6 | 8.26 | **2.62** | **1.61** | 2.14 | 2.66 | 1.43 |
| 3. IMMERSION IN 10 PER CENT SOLUTION OF SODIUM SULFATE. AVERAGES OF *B*, *D*, AND *E* MIXES. GROUPS OF LOW, MEDIUM AND HIGH RESISTANCE. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Low...... | 1,2,4,5,6,7,8,16 | 87.4 | 121 | 86 | 200 | 86 | 49 | 53 | 45 | 42.5 | 29.0 | **12.52** | 8.06 | 3.04 | **0.95** | 71.5 | 9.07 | **2.37** | **2.42** | 2.10 | 2.62 | 1.14 |
| Medium.. | 3, 12, 15, 18... | 170.8 | 128 | 80 | 150 | 137 | 61 | 53 | 41 | 48.2 | 24.9 | **9.25** | 9.95 | 3.02 | **0.87** | 73.1 | 8.86 | **2.41** | **1.72** | 2.13 | 2.85 | 1.02 |
| High...... | 9, 10, 11, 13, 14, 17.......... | 281.0 | 109 | 79 | 287 | 107 | 56 | 59 | 47 | 47.9 | 25.7 | **8.02** | 9.53 | 3.07 | **1.37** | 73.6 | 8.17 | **2.65** | **1.62** | 2.15 | 2.66 | 1.51 |
| 4. FREEZING AND THAWING, *H* CYCLE, 30 PER CENT LOSS, 28 DAYS. AVERAGE OF *B*, *D*, AND *E* MIXES. GROUPS OF LOW, MEDIUM AND HIGH RESISTANCE. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Low...... | 1, 3, 5, 10..... | 158.2 | 94 | 74.5 | 223.8 | 42.5 | 48 | 49 | 20.3 | 41.2 | 30.0 | 10.27 | **9.52** | 3.35 | 0.80 | 71.2 | 9.00 | 2.41 | **1.84** | 2.08 | 2.59 | 1.20 |
| Medium.. | 4, 9, 11, 13, 14, 16, 17, 18... | 222.4 | 111.2 | 80 | 235 | 132 | 57 | 59 | 46.5 | 48.5 | 24.8 | 9.20 | **9.28** | 2.99 | 1.20 | 73.3 | 8.46 | 2.54 | **1.81** | 2.15 | 2.67 | 1.37 |
| High...... | 2, 6, 7, 8, 12, 15. | 112.1 | 146.1 | 91 | 208 | 108 | .... | .... | 58.7 | 44.5 | 27.9 | 11.77 | **8.18** | 2.91 | 1.07 | 72.47 | 8.86 | 2.44 | **2.36** | 2.12 | 2.64 | 1.06 |
| 5. FREEZING AND THAWING, *H* CYCLE, 90 DAYS, 30 PER CENT LOSS. AVERAGE OF *B*, *D*, AND *E* MIXES. GROUPS OF LOW, MEDIUM AND HIGH RESISTANCE. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Low...... | 1,3,8,9,11,13,18 | 192 | 108 | 67 | 247 | 158 | 53 | 55 | 48 | 45.3 | 27.4 | **9.6** | **9.5** | 3.1 | 1.09 | 72.6 | 8.70 | 2.48 | **1.84** | 2.12 | 2.63 | 1.34 |
| Medium.. | 2, 5, 10, 14, 15, 16, 17...... | 160 | 117 | 85 | 208 | 64 | 59 | 50 | 38 | 46.7 | 26.2 | **9.8** | **9.1** | 3.0 | 0.97 | 73.0 | 8.61 | 2.52 | **1.88** | 2.13 | 2.65 | 1.12 |
| High...... | 4, 6, 7, 12...... | 101 | 142 | 104 | 230 | 79 | 57 | 62 | 52 | 44.0 | 27.7 | **12.5** | **7.8** | 2.9 | 1.22 | 74.2 | 8.97 | 2.39 | **2.50** | 2.13 | 2.66 | 1.24 |
| 6. FREEZING AND THAWING, *S* CYCLE, 28 DAYS, 5 PER CENT LOSS. *E* MIX. GROUPS OF LOW MEDIUM AND HIGH RESISTANCE. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Low...... | 1, 5, 15, 16, 17. | 152.1 | 110.2 | 80.4 | 138 | 50.2 | 53.7 | 48 | 36.2 | 45.6 | 26.5 | 10.45 | 9.84 | 2.92 | 0.68 | 72.1 | 7.46 | 2.31 | 1.85 | 2.09 | 2.63 | 1.10 |
| Medium.. | 2,3,4,6,7,9,14 | 137.4 | 122.6 | 89.6 | 247.5 | 68.1 | 55.6 | 59.6 | 39.7 | 43.3 | 28.6 | 11.29 | 8.08 | 3.09 | 1.16 | 71.9 | 8.52 | 2.50 | 2.15 | 2.12 | 2.63 | 1.31 |
| High...... | 8, 10, 11, 12, 13, 18.......... | 224 | 122.0 | 76.0 | 357 | 191 | 57 | 56.5 | 57.65 | 48.05 | 25.55 | **8.98** | 9.28 | 3.10 | 1.30 | 73.6 | 8.39 | 2.59 | 1.79 | 2.15 | 2.66 | 1.26 |
| 7. FREEZING AND THAWING, *S* CYCLE, 90 DAYS, 5 PER CENT LOSS. *E* MIX. GROUPS OF LOW, MEDIUM AND HIGH RESISTANCE. |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Low...... | 1,3,5,6,7,15,16 | 110.1 | 118.2 | 84.3 | 151 | 38.14 | 54 | 52.7 | 33.84 | 43.4 | 27.38 | 11.90 | 9.12 | 3.10 | 0.66 | 70.75 | 9.42 | 2.24 | 2.17 | 2.09 | 2.63 | 1.09 |
| Medium.. | 2,4,14,9,10,11, 17 ..... | 216.2 | 114 | 85.6 | 175.5 | 86 | 56.3 | 71 | 41.42 | 45.86 | 27.8 | 8.90 | 8.64 | 3.04 | 1.35 | 73.6 | 8.02 | 2.72 | 1.84 | 2.14 | 2.64 | 1.45 |
| High | 8, 12, 13, 18 | 195.6 | 129 | 74 | .... | 251.5 | 57 | 54 | 69.0 | 48.85 | 24.92 | 9.90 | 9.28 | 2.95 | 1.30 | 73.8 | 8.56 | 2.46 | 1.90 | 2.16 | 2.68 | 1.12 |

or a combination of $C_3A$ and $C_4AF$. Figure 10 (*b*) shows the relation obtained. The plot of $C_3A$ alone shows a relationship, but it was not considered as good as that given by $2C_3A - C_4AF$. Apparently high values of the quantity $2C_3A - C_4AF$ accompany low resistances and *vice versa*. Unfortunately the cements separate into two groups. The average values for these two groups given in Table III, section 1, indicate no significant difference between the groups. No explanation of their occurrence has been obtained to date. In this connection it is well to point out that if certain groups of cements be selected, it is not at all difficult to obtain very good relations between various quantities. Since different relations can be obtained by selecting various groups of the cements of this investigation, it follows that if a larger number of cements or a different group of the same number were used then relations appreciably different from those shown in this paper might be obtained. In other words, the relations shown should be regarded as applying to the 28 cements of this investigation and more particularly to the 18 portland cements until further investi-

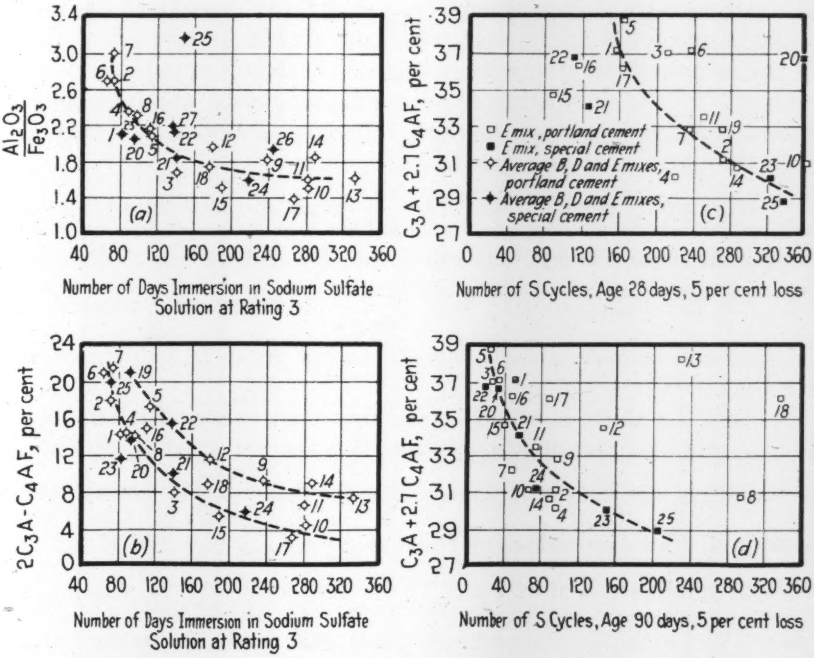

FIG. 10.—Constitution *versus* Resistance to 10 per cent Sodium Sulfate and the *S* Cycle.

gation with other cements has shown the relations to be of wider significance or not to hold at all.

Separating the 18 portland cements into three groups of low, medium and high resistance to sodium sulfate gave the data of Table III, section 3. It will be seen from the bold-faced values that an increase in resistance is accompanied by a decrease in tricalcium aluminate. No cement in the low-resistant group had less than 10.8 per cent tricalcium aluminate, nor did any cement of the high-resistant group have a value of tricalcium aluminate greater than 9.1. In the intermediate group the values of tricalcium aluminate ranged from 8.1 to 10.3 indicating some overlapping with the high group. When such overlapping is marked, differences between the averages of the groups cannot be regarded as significant.

Overlapping occurred with the $\frac{SiO_2}{R_2O_3}$ ratio on the low and medium group, but there was only one value, that for cement No. 13, less than 2.59 for the high group. With $\frac{Al_2O_3}{Fe_2O_3}$, overlapping occurred on the high and medium groups, but on the low group no value occurred less than 2.08.

From the above it would appear that high values of resistance to 10 per cent sodium sulfate solution may be expected if tricalcium aluminate is less than 9 per cent and if the ratio $\frac{SiO_2}{R_2O_3}$ is greater than 2.50; and that low values of resistance may be expected if the tricalcium aluminate is in excess of 10 per cent and $\frac{Al_2O_3}{Fe_2O_3}$ is greater than 2.00. While the limiting values given here probably apply to the cements and test procedure of this investigation only, the statement may be generalized by stating that high resistance to 10 per cent sodium sulfate solution will be probable if the percentage of tricalcium aluminate is low and the ratio $\frac{SiO_2}{R_2O_3}$ is high, and that low resistance is probable if the percentage of tricalcium aluminate is high and the ratio $\frac{Al_2O_3}{Fe_2O_3}$ is high. This does not mean that cements that do not comply with these limits may not be either low or high; for example, cement No. 13 with a value of $\frac{SiO_2}{R_2O_3}$ of 2.32 is in the high group.

The relation of values of the ratio $\frac{Al_2O_3}{Fe_2O_3}$ to the resistance to

10 per cent sodium sulfate are shown in Fig. 10 (*a*). There is a somewhat similar relationship with the ratio $\frac{SiO_2}{R_2O_3}$.

Another interesting fact was brought out by examination of the sodium sulfate tests. For one group of cements, group No. 1, the resistance to sodium sulfate solution varies with the water-cement ratio; for another, group No. 2, the resistance is independent of the water-cement ratio. One cement of the latter group, No. 9, is shown in Fig. 3. The remaining cements of the group belong to group No. 1 when visual rating is used. Judged by loss in weight, cement No. 3 places in group No. 2.

The average characteristics of these two groups is shown in Table III, section 2. The close agreement between the cements included in groups Nos. 1 and 2 of Table III, section 2, and the low and high resistance groups of Table III, section 3, should be noted. Since the cements of group No. 1 are low in resistance relative to group No. 2 it is to be expected from the previous discussion that the average tricalcium aluminate and $\frac{Al_2O_3}{R_2O_3}$ will be high and the average $\frac{SiO_2}{R_2O_3}$ will be low relative to the corresponding values of the cements of group No. 2. No other significant variation between the averages appears other than that the average resistance of the *E* mix is somewhat greater in the *S* cycle.

The radically different behavior of these two groups with change in water-cement ratio indicates clearly that the water-cement ratio must be considered in rating cements relative to their resistance to sodium sulfate. This is illustrated in the following tabulation for the 18 portland cements arranged in order from least to more resistant based on visual rating:

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Average, *B*, *D* and *E* mixes.. | 6 | 2 | 7 | 1 | 4 | 8 | 16 | 5 | 3 | 18 | 12 | 15 | 9 | 17 | 11 | 10 | 14 | 13 |
| *B* Mix... | 6 | 7 | 1 | 2 | 4 | 8 | 15 | 16 | 5 | 3 | 18 | 9 | 14 | 11 | 17 | 10 | 13 | 12 |
| *D* Mix... | 6 | 2 | 7 | 1 | 4 | 8 | 5 | 16 | 3 | 12 | 15 | 18 | 9 | 17 | 11 | 10 | 14 | 13 |
| *E* Mix... | 2 | 6 | 4 | 7 | 16 | 8 | 1 | 5 | 12 | 3 | 18 | 15 | 9 | 17 | 10 | 11 | 14 | 13 |

The average change from low to high position is 2.4, with a minimum change of 0 for cement No. 8 and a maximum change of 9 for cement No. 12.

It is interesting to note from Table III, sections 2 and 3, that the percentage of uncombined lime is higher for the cements of the high-resistant groups and that tricalcium silicate is low for the low-resistant groups. While no special importance is attached to the above by the author, they do indicate that an increase in uncombined and liberated lime may not be so important as is sometimes stated.

*Relations Between Constitution and Freezing and Thawing:*

Referring to Table II, it will be seen that the coefficient of $C_4AF$ is heavily minus for the *H* and *S* cycle equations, while for the *F* cycles, it is minus for the 28-day equation and plus for the 90-day equation. Since data for only 9 cements were available for the *F* cycle, the coefficients for this cycle are not given as much weight as those for the *H* and *S* cycles.

In the equations for the individual mixes (not shown in Table II) small plus coefficients for $C_4AF$ and small negative coefficients for dicalcium silicate were obtained for the *B* and *E* mixes of the *H* cycle 28-day specimens and for the *B* mix of the *F* cycle 28-day specimens. Small negative coefficients were also obtained for tricalcium aluminate in the equations for the *D* and *E* mixes of the 90-day *H* cycle, the *D* mixes of the 28 and 90-day *F* cycles. As is shown in Table II rather large negative coefficients of tricalcium aluminate were obtained in the two equations for the *E* mixes in both the 28-day and 90-day *S* cycles. In no case was a negative coefficient found for $C_3S$ which indicates strongly that the resistance of mortars to artificial freezing and thawing does not decrease with increase in tricalcium silicate as has been stated to be the case for concrete in service.[1]

Due to the misinterpretation of a chance relation in the data on 8 cements used in the preliminary series of tests, the author started the investigation reported on in this paper with the belief that the data would show that the resistance to freezing and thawing decreased with increase in tricalcium silicate. However, as the evidence accumulated that tricalcium silicate was not detrimental either to resistance to freezing and thawing or to sodium sulfate, the preliminary data was reexamined and found to be in agreement with the data of the present paper.

As in the sodium sulfate tests, averages for the cements of low, medium and high resistance to freezing and thawing were calculated for the freezing-and-thawing cycles.

Referring to Table III, section 4, it will be observed that the average $C_4AF$ decreases as the resistance to the 28-day *H* cycle increases. While this relation is in accordance with the least-square solution, the overlapping of individual values leaves considerable doubt as to its significance. The relatively high average value of the ratio $\frac{Al_2O_3}{Fe_2O_3}$ for the high resistant group indicates the probability of high resistance to this cycle if the ratio $\frac{Al_2O_3}{Fe_2O_3}$ is high.

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[1] Ira Paul, "Concrete Pavement Failures Due to High Lime Cement," *Engineering News-Record*, February 16, 1933, p. 213.

In Table III, section 5, the high value of $C_3A$ for high resistance is regarded as of no significance because of overlapping of individual values. Again the trend in $C_4AF$ is marred by overlapping values. The average value of the ratio $\frac{Al_2O_3}{Fe_2O_3}$ is high for the high-resistant group. In the 28-day and 90-day *S* cycle group averages, Table III, section 6 and 7, there appears to be little of any significance. No group averages are shown for the *F* cycle because of the small number of portland cements in this cycle.

Since the numbers designating the cements are in order of the tricalcium silicate percentage, increasing with increase in tricalcium silicate, within the portland and special cement groups, respectively, it is evident from following the numbers of the plotted points in any figure showing freezing-and-thawing data or the groupings of Table III, sections 4 to 7, inclusive, under the heading "Cement Numbers," that there is no obvious relation between tricalcium silicate and resistance to freezing and thawing.

The relation between constitution and the resistance to freezing and thawing appears to be somewhat vague. However when single mixes of a given cycle are considered alone, a relation of sorts may be obtained as is illustrated by Fig. 10 (*c*) and (*d*). It may be that some more general relation will develop on further study of the completed data.

## CONCLUSIONS

The following conclusions have been drawn. Since the data are not yet complete, some of these must be regarded as tentative only:

1. The absorption of mortars of equal flow is a function of the water content ($w_m$) and does not vary with the water-cement ratio.
2. The resistance to artificial freezing and thawing can be expressed as a function of the $\frac{c}{v+c}$ ratio.
3. The brand of cement markedly affects the resistance of mortars of equal water-cement ratio to artificial freezing-and-thawing

cycles. The spread between the high and low resistances varies with the type of cycle. In this investigation, it is greatest for the *S* cycle and least for the *F* cycle.

4. Conclusions drawn from artificial freezing-and-thawing cycles should be applied with caution to natural weathering conditions until definite correlation of the artificial cycle with natural weathering is established.

5. A period of air hardening increases the resistance of mortars to artificial freezing and thawing more than a longer period of moist curing, the increase varying with the cycle used and also with the cement. Oven drying decreases the resistance to freezing and thawing.

6. For a given cement or for the average of a number of cements, the resistance of mortars to artificial freezing and thawing increases with increase in compressive strength, but for mortars of the same water-cement ratios and consistency made from different cements, this relation does not hold and for high water-cement ratios may be reversed.

7. The relation between the resistance of mortars to 10 per cent sodium sulfate solution and compressive strength varies with the cement. For some, in general those of low resistance to sodium sulfate, the resistance increases with increase in compressive strength while for others, in general those of high resistance to sodium sulfate, the resistance appears to be independent of the compressive strength.

8. The resistance to 10 per cent sodium sulfate solution varies with the water-cement ratio for some cements, while the resistance of other cements is independent of the water-cement ratio. In general, the first group are low and the second group are high in resistance to sodium sulfate.

9. There is no relation between the resistance to 10 per cent sodium sulfate solution and the resistance to the three artificial cycles of freezing and thawing.

10. There is no significant relation between Merriman's index of disintegration and the resistance to 10 per cent sodium sulfate solution nor between Merriman's index and the resistance to the three artificial freezing-and-thawing cycles of this investigation.

11. Within the narrow range of porosities of this investigation, the resistance to artificial freezing and thawing is independent of the porosity.

12. From the data on the cements of this investigation, it appears that high resistance to 10 per cent sodium sulfate solution is probable if the percentage of $C_3A$ is low and the ratio $\frac{SiO_2}{R_2O_3}$ is high,

and that low resistance is probable if the percentage of $C_3A$ is high and the ratio $\frac{Al_2O_3}{Fe_2O_3}$ is high.

13. The evidence so far obtained on the resistance of mortars to artificial freezing and thawing indicates that $C_3S$ is the most desirable and $C_4AF$ the least desirable of the four major constituents.

*Acknowledgment:*

The author is indebted to the Graduate School, Dr. Guy Stanton Ford, Dean; to the Engineering Experiment Station, Dean Ora M. Leland, Director; to the Civil Engineering Department, Professor F. H. Bass, Chairman; and to the Structural Division, Professor J. I. Parcel, Chief; for funds required for this investigation. The author is indebted also to numerous cement companies for chemical analyses and to the Minnesota State Highway Department for specific gravity tests on the cements.

# APPARATUS FOR FABRICATING AND TESTING MASS-CONCRETE CYLINDERS

By R. F. Blanks[1] and E. N. Vidal[2]

## Synopsis

This paper describes equipment and apparatus for fabricating and testing mass concrete cylinders of various sizes up to 36 in. in diameter in a comprehensive program of mass-concrete research for Boulder Dam. Special methods of laboratory procedure developed for conducting large cylinder tests are discussed and test results indicating the degree of uniformity secured are presented briefly in comparison with those obtained in usual series of laboratory test programs.

## Introduction

The program of research for Boulder Dam being conducted by the U. S. Bureau of Reclamation involves mass concrete tests to determine the effects of maximum size of aggregate, varying mix proportions, size of test specimen and other related factors in cylinders varying from 2 in. to 36 in. in diameter. The usual laboratory equipment and methods were manifestly inadequate for the large-scale production of concrete test specimens, necessitating the development of original and specially adapted apparatus and testing procedure. A number of preliminary problems, each of which required quite extensive research programs, were solved before proceeding with the main series of tests.

Approximately one year's time of a large laboratory personnel was required in the development and preliminary work. Such features as machine mixing, vibratory compaction, wet screening, grinding of cylinder ends in lieu of capping, special curing control and uniformly consistent conditions of testing for compressive strength, elastic properties and permeability constitute some of the major departures from the customary standards of laboratory work in concrete testing.

The mass-concrete tests require approximately twelve carloads of aggregates and 300 bbl. of cement. The aggregates are separated into twelve sizes by hand-screening through standard sieves at the Arizona gravel deposit, the source of supply for Boulder Dam. The separated materials are shipped in 100-lb. sacks to the Denver laboratories where the tests are being conducted. A quantity of cement sufficient to meet the requirements of the entire research program was purchased in one lot, thoroughly blended and placed in air-tight storage to be used as needed.

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### APPARATUS AND EQUIPMENT

*Storage Facilities.*—Wood bins lined with No. 24 gage galvanized sheet metal are used for storing current supplies of the different sizes of aggregate which vary from cobbles retained on a 6-in. screen to fine sand passing the No. 100 sieve. Features of the design of these bins include open tops for convenience in filling either from trucks or with an overhead trolley-block crane; hopper bottoms, adjustable to conform to the angle of repose of the various sizes; and hinged front panels to facilitate removal of the materials with shovels.[1] The total bin capacity is about ten cubic yards of sand, gravel and cobbles, sufficient to cast six 36 by 72-in. cylinders.

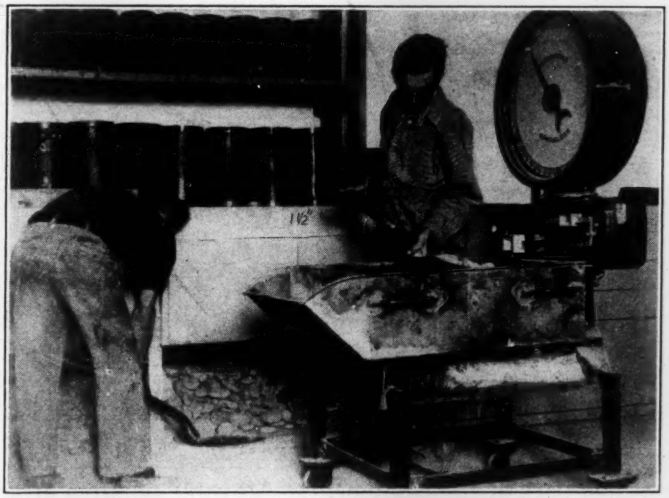

FIG. 1.—Weighing Aggregate Batch.

Standard 55-gal. steel drums are employed for cement storage. These drums have 9-in. end openings fitted with lock covers to permit air-tight sealing. The filled drums are handled by means of trolley-block cranes.

*Rolling Equipment.*—A standard steel-body motor truck having a capacity of 1½ tons is used in transporting materials, supplies, equipment and debris. The rolling-stock used in the laboratory proper includes two-wheel warehouse trucks, four-wheel platform and wagon trucks, dollies, wheel-barrows and concrete buggies. The large cylinders, after removal from the molds, are transported by means of a specially designed chair crane mounted on heavy casters. An overhead track extending to all parts of the laboratory provides for lifting and moving awkward and heavy objects with a 1-ton traveling trolley block.

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[1] Two of these bins are shown in the background of Fig. 1.

*Scales.*—Dial-indicating platform scales (Fig. 1) mounted on wheels for moving along in front of the bins in weighing batch aggregates or to various convenient points in the laboratory form an indispensable part of the equipment. Movable pointers attached to the outer edge of the dials of these scales greatly facilitate weighing successive sizes of aggregates for individual batches of concrete. A floor-type platform scale equipped with a dial indicator and having a total capacity of 10,000 lb. is used in determining the weight of large cylinders, thereby providing reliable data on the unit weight of mass concrete. A small platform scale with a beam indicator graduated to 0.01 lb. is available for weighing cement and other materials or objects where a high degree of accuracy is required.

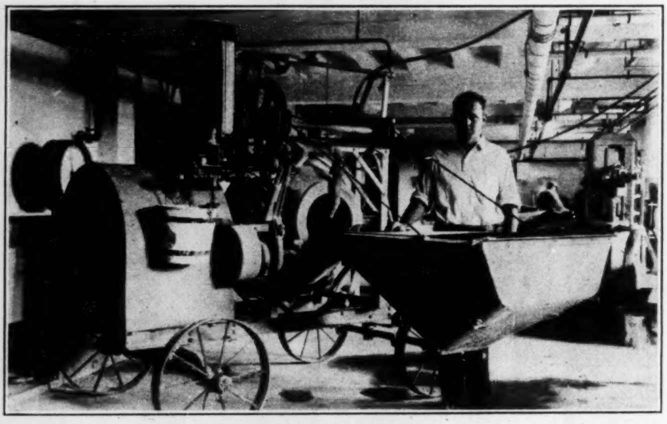

FIG. 2.—Mixer.

*Hoppers.*—Hoppers and buckets of various sizes and shapes were specially fabricated of No. 14 gage sheet iron with reinforced edges to meet particular requirements in batching and handling wet concrete. Suitable handles and chain slings permit lifting either by hand or by hoists.

*Concrete Mixer.*—The machine chosen for the laboratory mixing of concrete is a commercial unit of 3½ cu. ft. capacity (Fig. 2). This mixer is a tilting-drum type, driven by a 2-hp. electric motor and is equipped with a power loading skip. One of the principal factors governing the choice of this particular machine is the double cone shape of the drum and the blade arrangement.

Particular attention is given to the accurate control of water for each batch of concrete. A supply of water for use in mixing is maintained at a constant temperature of 70 F. (21 C.) by storage in a 125-gal. tank suspended from the laboratory ceiling. This tank is emptied each night and filled with fresh water each morning. A special copper tank conveniently attached to the mixer is filled through a hose and valve connection from the main storage tank at the start of mixing operations for each batch. This tank is provided with an overflow weir and a gage glass graduated to 0.1 lb. of water to insure accurate control of the quantity allowed to enter the mixing chamber through suitable valves and piping.

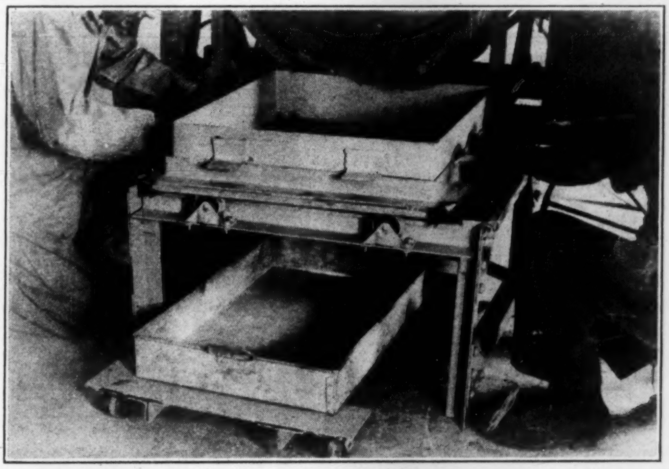

FIG. 3.—Wet-Concrete Screen.

*Screens.*—The apparatus for screening wet concrete containing cobbles to the maximum size allowable in 6-in. and 8-in. diameter specimens was designed and built specially for this purpose. Its dimensions are such that the mixer dumps directly on the screen (Fig. 3) and by manual operation the desired material is passed through to a hopper below, the latter being supported on a steel dolly for convenience in moving. A complete set of interchangeable screens is available for separating to various maximum sizes of aggregate. A gyratory riddle equipped with a full set of standard Tyler sieves, 20 in. in diameter, is used for separating large amounts of dry aggregates.

*Molds and Base Plates.*—A large variety of molds are required, from a minimum diameter of 2 in. to a maximum of 36 in. with height equal to twice the diameter in all cases. Figure 4 is a view of all the molds except the 2-in. which are standard type cast bronze in groups of three, set on glass plates. The 3-in. sizes are fabricated from steel tubing and are used in groups of three on a machined plate. The 6- and 8-in. molds are cast iron, bored to close tolerances, and are equipped with machined base plates which are fastened to the molds by capscrews. This expedient prevents loss of water and has proved to be a great convenience not only in handling but also in using the vibrators for compacting the concrete, which creates a tendency for the molds to "walk" off the base plate.

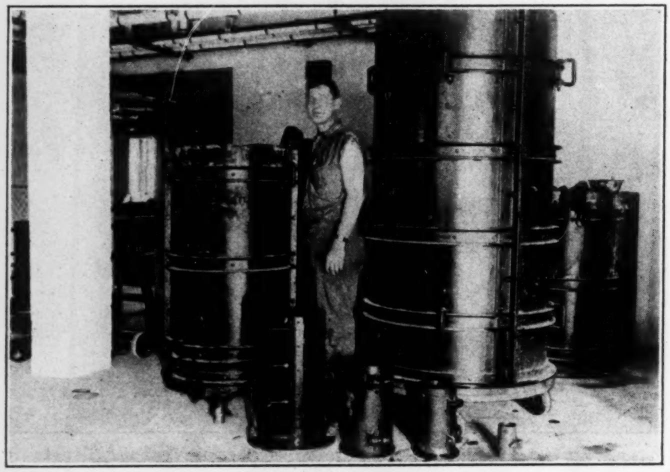

FIG. 4.—Cylinder Molds, 36 by 72 in. to 3 by 6 in.

The molds from 12 to 36 in. in diameter are of mild-steel plate carefully rolled and reinforced, when required, by steel channels and angles. The 12 by 24-in. mold is in one piece with no reinforcement, and clamps to a machined plate by means of turnbuckle hook rods. The larger sizes are in two symmetrical sections with flanged-angle longitudinal joints bolted together at about 4-in. centers. These molds are assembled on movable cast-iron base plates and clamped thereto by C clamps. The base plates are each equipped with three casters of the proper capacity to aid in handling and moving the filled molds. A filled 36 by 72-in. mold on its base plate weighs slightly more than four tons.

All of the structural steel molds are drilled at accurately measured points for casting brass inserts to which the elasticity apparatus is attached. The inserts are attached by means of capscrews, and rubber washers are placed between the insert and the mold to prevent water and mortar leakage. The capscrews are removed some six to eight hours after casting to eliminate shrinkage or settlement cracks around the inserts.

All molds are thoroughly coated with a thin film of graphite grease before use. Numerous experiments have established this material as the most suitable, not only from a standpoint of efficiency in preventing the concrete from adhering to the metal, but also because of its relative cleanliness and ease of application. Rubber or asbestos gaskets are used at the joints of the large molds to minimize water leakage to negligible quantities. A half-ton trolley-block, suitably located on an overhead rail, aids in the assembly of the large molds.

After removal, the molds are cleaned dry and then scrubbed with gasoline in which a small part of light oil has been mixed. The gasoline acts as a cleansing fluid and the oil adheres to the metal and reduces rusting. Suitable racks and storage spaces are provided in the laboratory for the molds when not in use.

*Vibrators.*—Various types of vibrators for compacting concrete were tried during the preliminary stages of the research program. Surface vibration, vibration equipment attached to the exterior of the molds, and others which transmitted the impulses to the concrete by the insertion of a vibrating rod were all investigated. The latter was found to be the most efficient and suitable for use where a large range in cylinder size was involved.

The vibrator finally adopted is of the internal type and consists of a steel tube housing an eccentric rotating element connected to a high-speed motor by means of a flexible shaft. The shaft is 10 ft. long and permits considerable freedom of movement of the vibrator itself. Variations in the amplitude and frequency of the vibratory impulses are accomplished by a rheostat control of the power line to the motor.

*Curing Room Air-Conditioning and Automatic Control Units.*—The laboratory is well equipped with curing facilities, there being two large curing rooms on the same level and two small rooms in a sub-basement, accessible by means of a hand-operated elevator. All curing rooms are automatically controlled for temperature and humidity by an elaborate system of apparatus which has individual air-

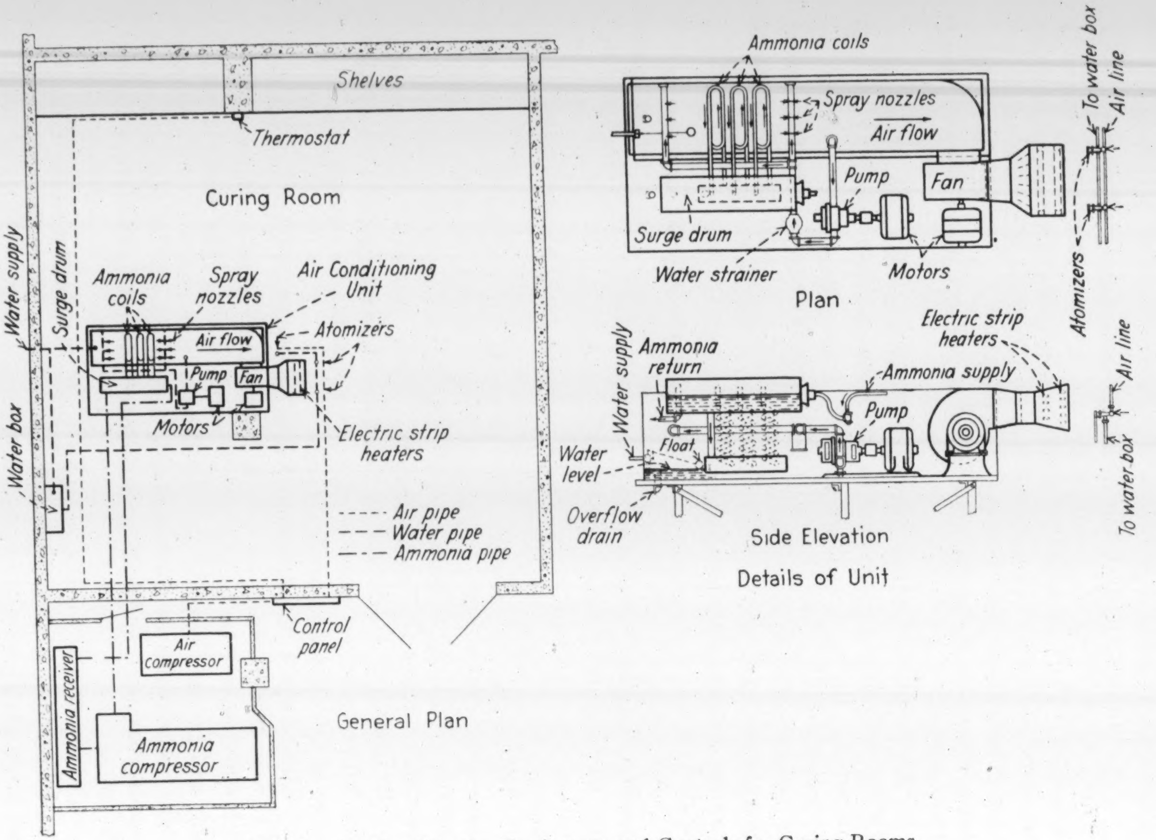
Shelves
Thermostat
Curing Room
Water supply
Surge drum
Ammonia coils
Spray nozzles
Air Conditioning Unit
Atomizers
Air flow
Pump
Fan
Motors
Electric strip heaters
Water box
Air pipe
Water pipe
Ammonia pipe
Control panel
Air compressor
Ammonia receiver
Ammonia compressor
General Plan
Ammonia coils
Spray nozzles
Air flow
Pump
Fan
Surge drum
Water strainer
Motors
Plan
To water box
Air line
Atomizers
Electric strip heaters
Ammonia return
Ammonia supply
Water supply
Pump
Float
Water level
Overflow drain
Air line
To water box
Side Elevation
Details of Unit

FIG. 5.—Air-Conditioning Equipment and Controls for Curing Rooms.

conditioning units in each room (Fig. 5). The individual units were specially designed and built for the purpose to fit the requirements of this particular installation. Temperature control to ±2 F. (±1 C.) is obtained by means of ammonia refrigerating coils and electric-heater strips over which a current of water-sprayed air is circulated. Heat is added or withdrawn as required by a thermostatically controlled compressed-air line which actuates the proper one of two pressure relays, adding heat if needed or allowing ammonia to expand through the coils if the room temperature is too high. The compressed air and ammonia are furnished to all the rooms from a centrally located plant. All ammonia lines are equipped with solenoid valves so as to isolate the other curing rooms when control conditions change in any one.

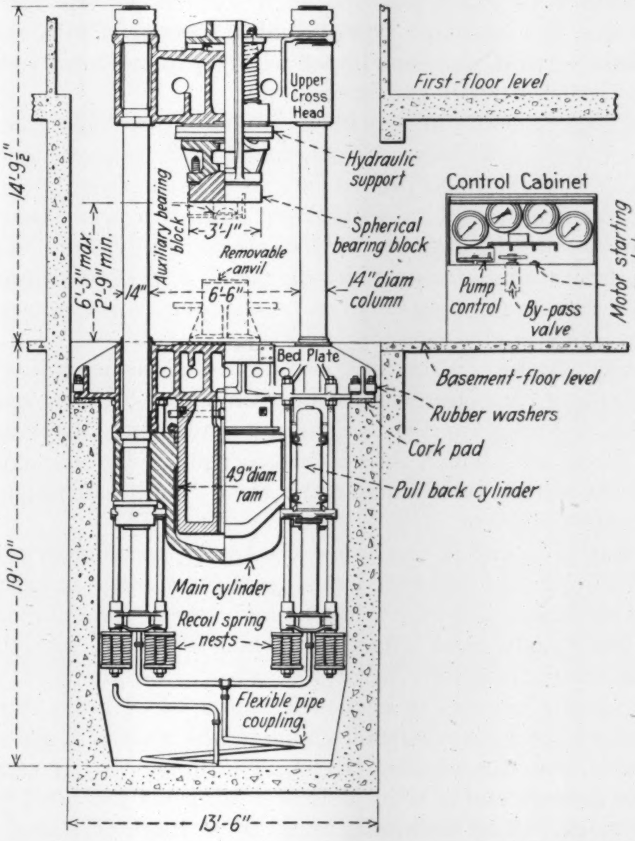
Upper Cross Head
First-floor level
Hydraulic support
Control Cabinet
14'-9½"
Auxiliary bearing block
Spherical bearing block
3'-1"
Motor starting switch
Removable anvil
6'-3" max.
2'-9" min.
14"
6'-6"
14" diam column
Pump control
By-pass valve
Bed Plate
Basement-floor level
Rubber washers
Cork pad
49" diam. ram
Pull back cylinder
19'-0"
Main cylinder
Recoil spring nests
Flexible pipe coupling
13'-6"

FIG. 6.—Sectional Elevation of 4,000,000-lb. Compression Testing Machine.

A dense fog is maintained in the rooms by means of compressed air atomizers, operating under a pressure of 60 lb. per sq. in. The number of atomizers used depends upon the size of the room, and they are located to provide optimum conditions.

*Grinding Machine.*—A commercial stone-polishing machine capable of operating with wheels up to 30 in. in diameter was installed in the laboratory to grind test specimen surfaces to smooth planes. The machine is a standard type, grinding specimens with the longitudinal axes vertical, and is equipped with a grinding head which may be moved both vertically and horizontally.

The large size of the specimens involved precluded the use of the end-surfacing machines ordinarily found in the concrete laboratory. The grinding machine has proved extremely satisfactory and cylinder ends are readily ground to within 0.002 in. of a true plane. The specimens are bedded in plaster of Paris (calcined gypsum) to provide a fairly level surface to start grinding and a No. 60 carborundum grain is used as the grinding medium. The ground surfaces are checked by means of a straight edge and feeler gage.

*Testing Machine.*—A 4,000,000-lb. hydraulic compression testing machine (Fig. 6) was purchased to accommodate the test specimens included in the program. By means of a movable anvil and auxiliary bearing blocks with which the machine is equipped, specimens 6 to 8 in. in diameter are tested under the same conditions which obtain for the larger cylinders.

As may be noted in the figure, the testing machine is essentially the same as others of this type but is greatly magnified in every detail and extends from a deep pit to the second floor of the building. The main cylinder, moving on a fixed ram which is integral with the lower platen, is actuated by the discharge of a rotary oil pump. The main cylinder casting extends to each side where bored holes engage the lower ends of the 14-in. diameter columns which support the movable crosshead above the working floor. The upper platen carries the hydraulic support and a 37-in. diameter hardened-tool-steel spherical bearing block. The hydraulic support is frictionless and of the diaphragm type, utilizing a light oil as the fluid medium to transmit the load to the indicating dials. There are four of these dials, recording simultaneously or individually as determined by suitable needle valves, and vary only in maximum capacity which is, respectively, 200,000, 1,000,000, 2,000,000 and 4,000,000 lb. The load is observed on the dial whose range is appropriate for the size of specimens under test.

The motor-driven rotary oil pump is designed to provide a variable discharge both in intensity and direction. Thus by controlling a single handwheel in the operating cabinet the rate of load application may be accurately governed at any desired value. A uniform rate of load application offered the most likely solution to the problem of providing uniform test conditions for a wide range of specimen sizes.

*Load Control.*—The load-rate instrument developed in the Denver laboratory consists essentially of a variable-speed disk-type motor which drives an auxiliary hand mounted directly in front of the load indicator of the dial gages. The auxiliary hand is driven at any desired uniform rate and the load indicator is made to follow by manipulation of the controls of the testing machine.

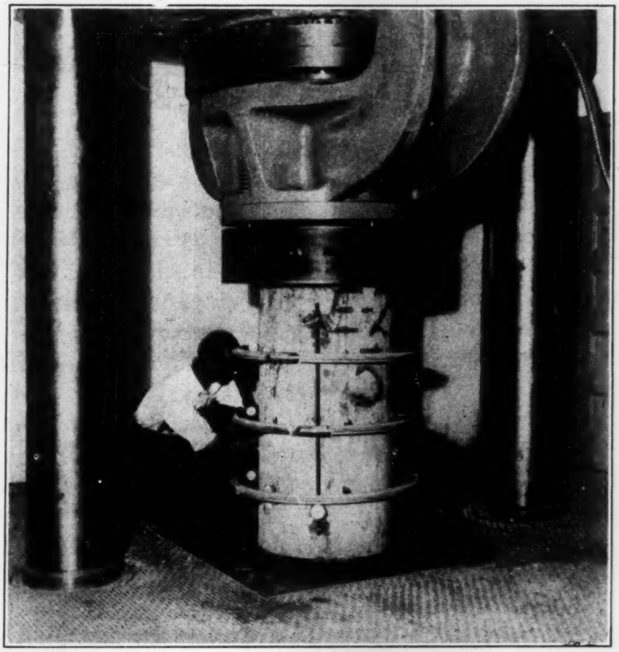

FIG. 7.—Large Cylinder Equipped for Strain Measurements Under Load.

The device now in use was constructed by remounting the operating mechanism of an ordinary house watt-hour meter and providing suitable gear ratios and pulley connections for driving the control hand. The current coils were rewound and variable rheostats connected to the load side of the meter. Thus, the speed of the load-rate instrument may be controlled to any desired rate depending upon the size of specimen under test and the rate of load application employed.

*Elasticity Apparatus.*—The apparatus for measuring the elastic deformations of test specimens under load was designed and constructed by the laboratory personnel after considerable time and effort had been spent investigating previous studies in this field. The devices finally adopted for the large specimens (Fig. 7) are composed of Invar steel strain gages for measuring the longitudinal strains and cast aluminum frames on which are mounted dial indicators graduated to 0.0001 in., for measuring the lateral strains. The longitudinal strain gages as used are mounted on the specimens by suitable grips which screw into the brass inserts cast in the concrete. Three are used on each cylinder and they are located at the third points around the circumference. The lateral frames are also three in number and are located at the quarter points. These are supported by ball bearings on brackets which screw into other inserts properly placed. Clamp-type extensometers and compressometers of rather conventional design but embodying several improvements over former models were designed and built for use on small specimens.

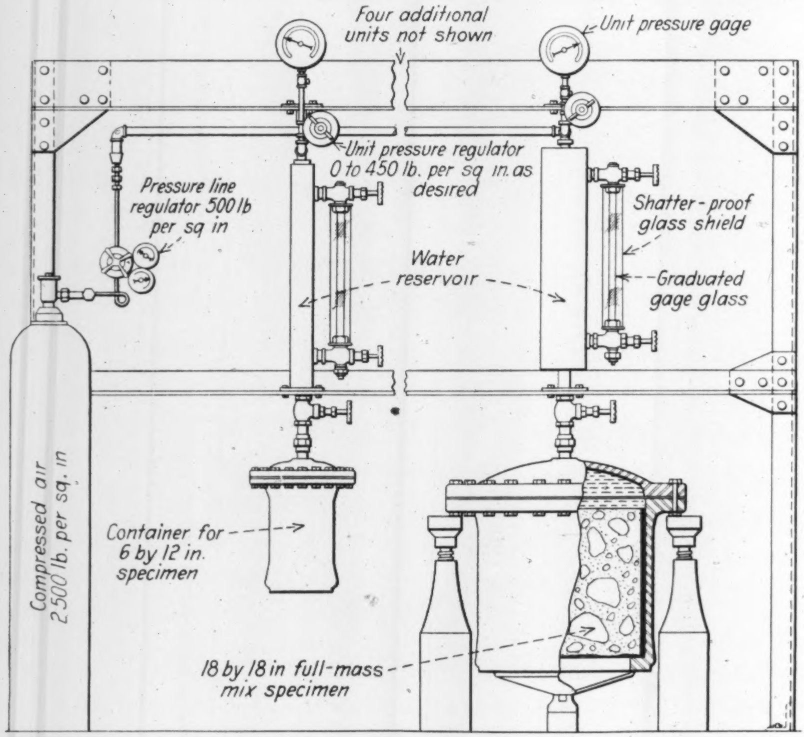
Four additional units not shown
Unit pressure gage
Unit pressure regulator 0 to 450 lb. per sq in. as desired
Pressure line regulator 500 lb per sq in
Shatter-proof glass shield
Water reservoir
Graduated gage glass
Compressed air 2500 lb. per sq. in
Container for 6 by 12 in. specimen
18 by 18 in full-mass mix specimen

Fig. 8.—Permeability Apparatus.

Calibration of the apparatus and of the dial indicators used therewith has required the design and construction of unique devices too complex to describe in this paper. All calibrations are based on a set of Johannson-Ford blocks.

*Permeability.*—Figure 8 is a diagrammatic view of the apparatus for testing the permeability of concretes and shows a large specimen under test. The figure is self-explanatory and the apparatus was designed to accommodate specimens from 6 by 6 in. to 18 by 18 in. in size. The large specimens when sealed in the cast-steel container weigh approximately 1500 lb. Galvanized iron containers of appropriate dimensions serve as molds for casting the test specimens which are cured for the desired periods in the 70 F. (21 C.) fog room.

## TEST METHODS

The various processes in the procedure for fabricating and testing concrete test specimens on a large scale as standardized in the Bureau of Reclamation laboratories may be discussed conveniently under five headings. The refinements of standardization include not only the major operations but also the minute details; however, it is possible to touch here upon only the more important phases and convey merely the general nature of the problems involved.

*Batching.*—Each size of aggregate including six separations of the sand, three of the gravel and two of the cobbles are weighed separately to insure correct and uniform proportioning of the individual batches of concrete. The complete batch of materials is placed in the loading skip with the coarser particles on the bottom, covered by the sand and the cement on top.

*Mixing.*—A primer batch of concrete is run through the machine at the start of all mixing operations. For each regular batch a portion of the water is introduced into the drum before dumping the loading skip. The cement and aggregates are dumped simultaneously with the introduction of the balance of the water required to complete the batch. Mixing is continued for 5 min. after all materials are in the drum. The nominal capacity of the machine is 3½ cu. ft.; however, the standard laboratory batch is limited to about 70 per cent of this amount to minimize spilling and to insure more uniform and thorough mixing. No trouble has been experienced in running cobbles up to 9 in. in size through the machine.

*Fabricating.*—Specimens are compacted by the vibrators in 6 to 9-in. lifts according to the size involved. The amount of vibration is of uniform duration for each lift as measured by a stop-watch and varies from 20 sec. for the small specimens to 45 sec. for the large ones. Six, eight, and twelve-inch diameter cylinders are filled with scoops, while end-dumping hoppers are used for the 18-in. and larger sizes.

*Curing.*—Specimens are placed in the curing room immediately after fabrication and remain therein for 24 hr., at which time the molds are removed. The large specimens are left on the movable plates for an additional 24 hr. after removal of the molds.

The curing rooms are equipped with suitable racks for specimens up to 16 in. in height. The racks are of heavy wood construction built along the walls so as to leave a large amount of open floor space in the center of the rooms for placing the larger cylinders. Specimens remain in the curing rooms until time of test, being removed only for surface grinding.

*Testing.*—From 1 to 7 days prior to the test date, the specimens are surface ground for test and are returned to the curing room to insure thorough saturation at the time of test. Some of the 6- and 8-in. sizes, especially those for test at ages up to seven days and those for compression test only, are sulfur-capped a few minutes before testing. This is accomplished by pouring a molten mixture of three parts of commercial powdered sulfur and one part of standard mill fire clay in a recess formed on the top of the specimens. A paper band or spring-steel ring forms the recess and a smooth surface is made by pressing on a machined plate while the capping material is still hot. The specimens may be tested immediately after capping and extensive tests have indicated that ground surfaces and sulfur caps provide equal strengths within one or two per cent.

Specimens are accurately centered in the testing machine and the load is applied at the uniform rate of 17 lb. per sq. in. per sec. The total load applied per second varies from 480 lb. for the 6-in. diameter to 17,300 lb. for the 36-in. specimens. Numerous tests, both in this laboratory and elsewhere, have justified this rate as sufficient for increasing the uniformity of test results for a program in which a wide range of specimen sizes are involved.

Elasticity measurements are made immediately after placing the specimens in the machine. The measurements are obtained as rapidly as possible; however, the procedure and technique employed is too involved to be described in this paper. A maximum lapse of 3 hr. for the largest specimens which are tested for elasticity occurs between removal from the curing room and the compression test and it has been observed that the concrete is dry for only a very thin section of the surface with the remainder saturated. Small specimens are returned to the curing room after elasticity tests to eliminate any reaction due to the possible drying out of the concrete. All other specimens, not tested for elasticity, are obviously in a saturated condition at time of test.

## UNIFORMITY OF TEST RESULTS

Preliminary series of tests established the sufficiency of equipment, apparatus and test methods in securing uniform test results before proceeding with the main program. The preliminary work showed individually and collectively the effects upon the uniformity of test results of the various special methods of procedure in comparison with results obtained by the usual methods. In all cases, development was continued until greater uniformity was obtained than was secured with present accepted standards.

TABLE I.—UNIFORMITY OF TEST RESULTS OBTAINED WITH MACHINE MIXING AND VIBRATORY COMPACTION IN COMPARISON WITH STANDARD HAND MIXING AND HAND RODDING.

| Age at Test, days | Average Mean Variations for Individual Daily Runs, per cent | | Day to Day Mean Variations for Entire Series, per cent | |
|---|---|---|---|---|
| | Machine Mixing | Standard Mixing | Machine Mixing | Standard Mixing |
| 7 | 3.5 | 3.6 | 3.9 | 3.0 |
| 14 | 3.2 | 3.8 | 3.3 | 1.9 |
| 28 | 3.0 | 3.0 | 3.3 | 2.4 |
| 90 | 3.6 | ... | 2.9 | ... |
| Average, all ages | 3.3 | 3.5 | 3.4 | 2.4 |

A comprehensive series of tests was made to compare the uniformity of results obtained by machine mixing and vibratory compaction with hand mixing and hand rodding in accordance with the Standard Methods of Making Compression Tests of Concrete (A.S.T.M. Designation: C 39 – 27).[1] Cylinders, 6 by 12 in., fabricated from 1½-in. maximum size aggregate concrete, capped with sulfur compound and tested at a uniform rate of load application, were used in both sets. The machine-mixed group involved four batches of concrete, twelve cylinders to one batch, made on each of eight different days in the laboratory with a consequent daily variation in temperature and humidity. Twelve cylinders from each day's run were broken at the ages of 7, 14, 28 and 90 days. The average mean variations for each day's run and the day to day mean variations are given in Table I.

The standard group consisted of nine single batches of concrete cast on six different days in a 70 F. (21 C.) fog room, maintained at constant temperature and humidity as described previously. Three cylinders from each day's run were tested at the ages of 7, 14 and 28 days. The corresponding uniformity figures are also shown in Table I. A total of 384 machine-mixed and 54 hand-mixed specimens were tested in the series.

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[1] 1930 Book of A.S.T.M. Standards, Part II, p. 143.

Even greater uniformity is obtained in the large cylinder tests. Seven groups of 36-in. diameter cylinders with aggregates up to 9 in., cast over a period of several months and tested at various ages showed an average mean variation of only 2.7 per cent. Corresponding groups of 24- and 18-in. specimens with 6- and 3-in. aggregates resulted in mean variations of 3.4 and 2.2 per cent, respectively.

Other series of tests were conducted to obtain a comparison between hand rodding and vibration; between grinding top surfaces of cylinders and various methods of capping; and between uniform loading and standard loading (0.05 in. head travel at idling speed). In all of these comparisons the usual standards gave slightly less uniformity of results than the new methods.

# A RAPID METHOD FOR THE DETERMINATION OF THE SPECIFIC SURFACE OF PORTLAND CEMENT[1]

By L. A. Wagner[2]

## Synopsis

An apparatus and method are described for making rapid determinations of the specific surface and also the particle size distribution of portland cement. The apparatus, which is essentially a turbidimeter, consists of a source of light of constant intensity which passes through a suspension of the cement in kerosine and then into a photo-electric cell. The current generated in the cell is measured with a microammeter and the readings afford a measure of the turbidity of the suspension. The relation between the turbidity of the suspension and the surface area of the suspended particles is calculated. Particle-size distribution is obtained by observing changes in turbidity as the particles settle from the suspension. Details of construction and operation of the apparatus are given in an appendix to the paper.

## Introduction

The inadequacy of sieves for measuring the fineness of portland cement has long been recognized. Several methods have been developed for determining sub-sieve particle sizes, but there is still urgent need for a rapid method suitable for use in industrial control laboratories.

An investigation was undertaken by the Cement Reference Laboratory at the U. S. Bureau of Standards to develop such a method. After a preliminary study of various methods, it was decided that the measurement of the turbidity[3] of cement in a liquid suspension offered the best means of achieving the results desired.[4]

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[1] Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate of the Cement Reference Laboratory at the U. S. Bureau of Standards, Washington, D. C.

[3] The turbidity of a suspension, as used in this paper, is defined as the logarithm of the ratio of the intensity, $I_0$, of the light transmitted by the clear suspending medium to the intensity, $I$, of the light transmitted by the suspension of the cement particles; that is, turbidity $= \log \frac{I_0}{I} = \log I_0 - \log I$.

[4] Some previous applications of the measurement of transmitted or scattered light for determining the size of small particles are described in the following publications:

G. F. A. Stutz and A. H. Pfund, "A Relative Method for Determining Particle Size of Pigments," *Industrial and Engineering Chemistry*, Vol. 19, No. 1, p. 51 (1927).

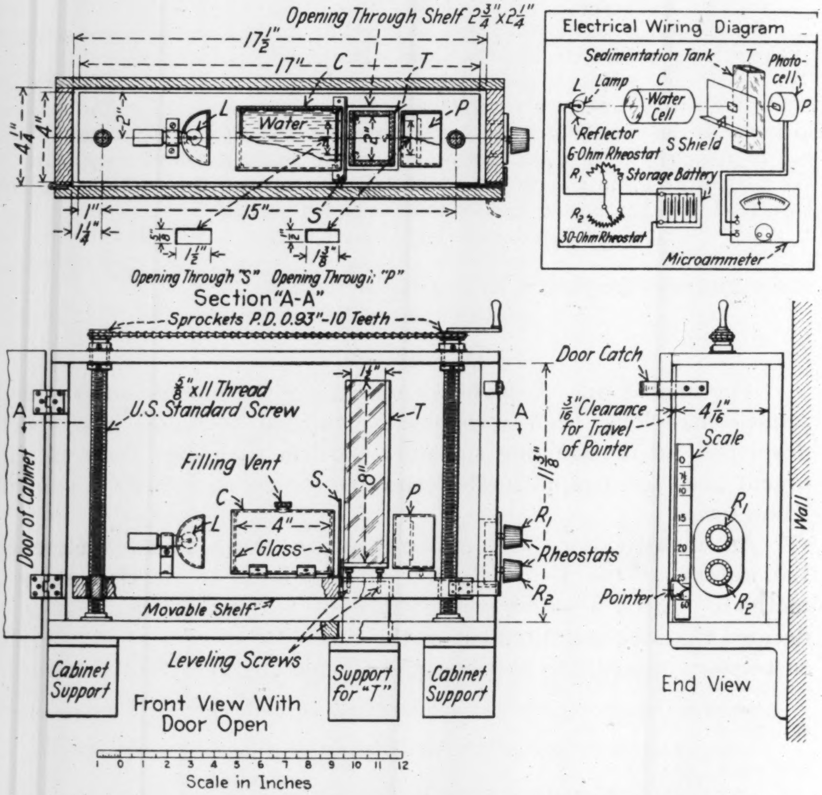
W. L. Gadd, "The Measurement of Small Particles," *Cement and Cement Manufacturing*, July, 1931, p. 763.

The Svedberg and Alfred J. Stamm, "The Use of Scattered Light in the Determination of the Distribution of Size of Particles in Emulsions," *Journal*, Am. Chemical Soc., June, 1925, p. 1582.

N. N. Andreev, "Apparatus for the Quantitative Investigation of Disperse Systems with Photo-Electric Cells," *Kolloid-Zeitschrift*, Vol. 57, p. 39 (1931).

N. N. Andreev, "Determination of Mean Sizes of Particles in Disperse Systems with the Aid of the Photo-Electric Cell," *Kolloid-Zeitschrift*, Vol. 57, p. 42 (1931).

An apparatus, herein called a turbidimeter, was constructed and a convenient method was developed for using the device in making determinations of the specific surface (square centimeters per gram) and particle size of fine-grained materials. The apparatus consists of a source of light of constant intensity, which light passes horizontally through a suspension of the cement into a photo-electric cell. The current generated in the photo-electric cell is measured with a microammeter, and the readings indicated afford a measure of the surface area of the suspended particles.


Opening Through Shelf 2¾"x2¼"
17½"
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Opening Through "S"
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Section "A-A"
Electrical Wiring Diagram
Sedimentation Tank
T
Photo-cell
P
L
Lamp
C
Water Cell
Reflector
6-Ohm Rheostat
S Shield
R1
Storage Battery
R2
30-Ohm Rheostat
Microammeter
Sprockets P.D. 0.93"-10 Teeth
⅝"x11 Thread
U.S. Standard Screw
A
A
1¼"
T
Door Catch
3/16" Clearance for Travel of Pointer
4 1/16"
Scale
11⅜"
8"
Filling Vent
S
C
P
L
4"
Glass
Door of Cabinet
R1
Rheostats
R2
Pointer
R1
R2
Wall
Movable Shelf
Leveling Screws
Cabinet Support
Support for "T"
Cabinet Support
Front View With Door Open
End View
1 0 1 2 3 4 5 6 7 8 9 10 11 12
Scale in Inches

FIG. 1.—Front and End Views, and Horizontal Section of the Turbidimeter.

The data recorded are the intensities of the light beam, in microamperes,[1] incident upon the photo-electric cell, and the settling time intervals at which the microammeter readings are taken. The settling time intervals are converted to particle sizes by means of Stokes' law and the conversion of the microammeter readings to surface area of the suspended particles is developed in this paper.

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[1] With the Weston Photronic cell and low resistance microammeter used, the current generated by the cell (microamperes) is directly proportional to the intensity of the light incident upon the cell. Hence the liberty is taken of expressing the relative light intensities in electrical units.

By these means the specific surface of a sample of cement and the distribution of that surface with particle size may be obtained. The size-weight distribution of the sample, if desired, may be calculated from the same data.

## APPARATUS AND METHOD OF OPERATION

Two modifications of the disappearing lamp-filament type of turbidimeter[1] were tried out but were discarded because of their lack of sensitivity. The readings were also subject to variation because of differences in the eyes of observers and because of eye fatigue of any one observer. Greatly increased sensitivity was attained, however, by replacing the eye with a photo-electric cell and measuring the intensity of a beam of light transmitted directly through a suspension of cement particles.

Referring to Fig. 1, the source of light, *L*, is a 6-candle-power electric lamp operated by a 6-v. storage battery. A parabolic reflector is mounted behind the lamp and is focused so that a beam of approximately parallel light passes through the glass settling tank, *T*, which contains a suspension of cement particles in kerosine. $R_1$ and $R_2$ are rheostats which serve to regulate the light to the desired intensity. A water cell, *C*, placed in the path of the beam absorbs the larger part of its radiant heat. The portion of the beam which is transmitted through the tank passes into a photo-electric cell, *P* (Weston Photronic type). The current generated in the photo-electric cell is measured with a microammeter and the reading indicated affords a measure of the turbidity of the suspension.

The lamp, water cell, and photo-electric cell are mounted on a shelf which is raised or lowered by two lead screws to bring the level of the light to any desired depth of the suspension in the settling tank. A pointer and scale on the outside of the cabinet indicates the position of the shelf.

The function of the movable shelf is to shorten the time required to obtain the sedimentation curve of a suspension. Turbidity readings of the larger particles, which fall out rapidly, are obtained at lowest level of the suspension (15 cm. in the present apparatus). Rather than wait for the smaller particles to settle to this level, which would require several hours, the shelf is raised so that the light beam is directed through lesser depths of the suspension. The levels at which the readings are made are selected so that about one minute elapses between readings for particle-diameter intervals of 5 microns. This gives the operator ample time to observe and record the microammeter reading, and to move the shelf into position for the next reading.

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[1] W. L. Gadd, "The Measurement of Small Particles," *Cement and Cement Manufacturing*, July, 1931, p. 763.

The sample to be tested is dispersed in kerosine, to which a few drops of oleic acid[1] have been added, by stirring in a test tube with a rotating brush. The mixture is then transferred to the settling tank and kerosine is added to bring the suspension to the desired volume. The tank is agitated to distribute the particles uniformly, and is placed in the path of the light beam. Microammeter readings are then observed at the proper depths and time intervals calculated from Stokes' law to correspond to particle diameters of 7.5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, and 60 microns.

Further details of construction and operation are given in the Appendix.[2]

### CALCULATIONS AND PRINCIPLES INVOLVED

The relation between size and rate of fall of spherical particles is given by the following form of Stokes' law:

$$t = \frac{18\, h\, u\, 10^8}{(P_1 - P_2) g\, d^2} \qquad \text{(1)}$$

where $t$ = time of settling in seconds,
$h$ = depth of suspension to level of light in centimeters,
$u$ = viscosity of dispersing medium in poises,
$P_1$ = density of particle in grams per cubic centimeter,
$P_2$ = density of dispersing medium in grams per cubic centimeter,
$g$ = gravity constant of acceleration, 980 cm. per sec. per sec., and
$d$ = diameter of particle in microns.

While it is convenient to indicate the size of an irregularly shaped particle by a diameter, it is necessary to define the latter term. For this paper the diameter of a particle is defined as the diameter, calculated from Stokes' law, of a sphere of the same density and falling velocity as the particle.

If the particles fall freely from a suspension, then, in accordance with Stokes' law, after a time interval, $t$, all particles larger than diameter, $d$, will have fallen below the level, $h$, of the light beam, and the change in intensity of the light transmitted through the suspension at this level will be due to the removal of these larger particles. The particles smaller than diameter $d$ that fall out of the path of the beam do not alter the intensity of the transmitted light because they are replaced by particles of a like size from upper layers of the suspension. That the change is due *only* to the removal of the larger particles has been clearly demonstrated by M. Kohn[1] in a graphic analysis of the fall of particles from a suspension. From these considerations it is seen that the microammeter readings observed at times of settling, $t_1$, $t_2$, $t_3$, etc., afford a measure of the surface area of the particles in the original suspension smaller than the corresponding diameters, $d_1$, $d_2$, $d_3$, etc., as calculated from Stokes' law.

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[1] Knowledge of the use of oleic acid as a deflocculating agent for cement in kerosine was obtained from Mr. Hubert Woods of the Riverside Cement Co., Riverside, Calif. Viscosity determinations showed no detectable change in the viscosity of the kerosine due to the addition of the oleic acid.

[2] See p. 566.

*Relation Between Intensity of Light Transmitted Through a Suspension and the Surface Area of the Suspended Particles:*

If a cement of known specific surface had been available, the relation between the intensity of light transmitted through a suspension and the surface area of the suspended particles could have been determined directly. However, since no such cement was available, the relationship was developed as described in the following paragraphs.

As the first step in this development the relation between turbidity and concentration was determined. To do this, turbidity readings were obtained at a constant depth and settling time for suspensions of increasing concentrations of the same cement. By this process the surface area of the suspended particles, although unknown, was increased in known definite ratios. The turbidity of these suspensions was found to vary with concentration, and, therefore, with surface area, as follows:

$$S_d = c(\log I_o - \log I_d) \qquad (2)$$

where $S_d$ = surface area, in square centimeters, of all particles in the sample smaller than $d$ microns in diameter;

$I_o$ = intensity of the light, in microamperes, transmitted through clear kerosine;

$I_d$ = intensity of the light, in microamperes, transmitted through the suspension at the time interval required for a particle, $d$ microns in diameter, to settle from the surface of the suspension to the center line of the light beam; and

$c$ = constant for each cement which will be called its transmittancy constant.[2]

A typical set of data from which Eq. 2 was derived is shown graphically in Fig. 2. Surface area of the particles is seen to be directly proportional to log $I_o$ − log $I_d$. Similar data were obtained for different cements and for different settling times and depths, and the above relation was found to hold good in all cases. With Eq. 2 established, the problem reduces to one of determining $c$ for any given cement.

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[1] M. Kohn, "Particle Size Determination by Means of Pipette Analysis," *Tonindustrie Zeitung*, No. 53, p. 730 (1929).

[2] $c$ is an arbitrary quantity which will depend upon the reflecting power, transparency, geometric form and size of the particles. For simplicity, $c$ is assumed to be independent of particle size.

In Fig. 3 is shown a curve plotted between $2 - \log I_d \left(= \frac{S_d}{c}\right)$ and particle size, $d$ (diameter), in microns. ($I_o$ is arbitrarily adjusted to 100 microamperes so that log $I_o$ − log $I_d$ is written $2 - \log I_d$.) In order to make these readings, the settling times, $t$, and depths of suspension, $h$, were arbitrarily selected by means of Stokes' law to correspond to values of $d$ = 7.5, 10, 15, 20, . . . 60 microns. Such a curve is designated as a particle size - surface area distribution curve.

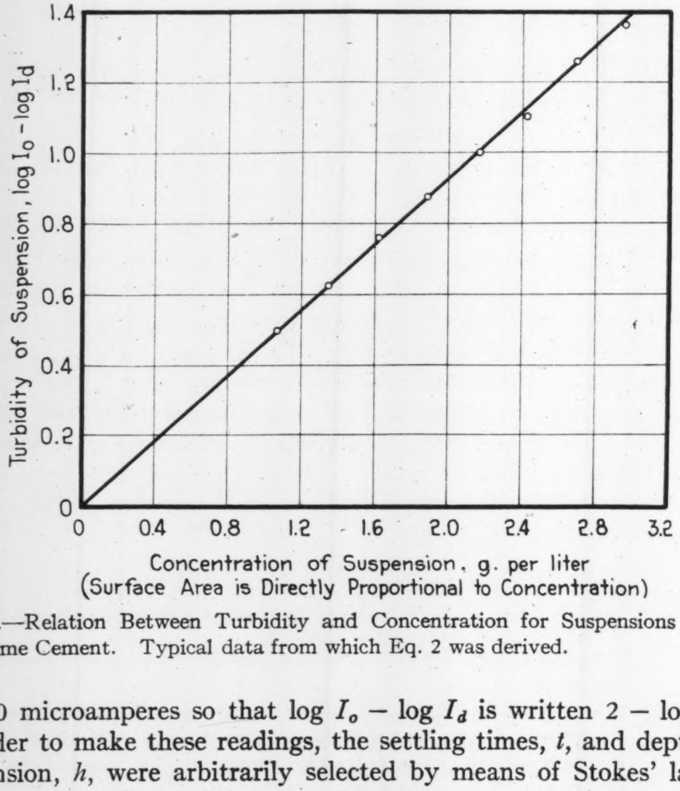

FIG. 2.—Relation Between Turbidity and Concentration for Suspensions of the Same Cement. Typical data from which Eq. 2 was derived.

Assuming $c$ to be constant for all sizes of particles in any given cement, the calculation of $c$ from the data of this curve, Fig. 3, is as follows: The relation of weight to surface area of spherical particles of equal size and having a specific gravity[1] of 3.15 is given numerically by Eq. 3:

$$w = 0.0000525\, Sd \qquad (3)$$

where $w$ = weight of the particles in grams,
$S$ = surface area of the particles in square centimeters, and
$d$ = diameter of the particles in microns.

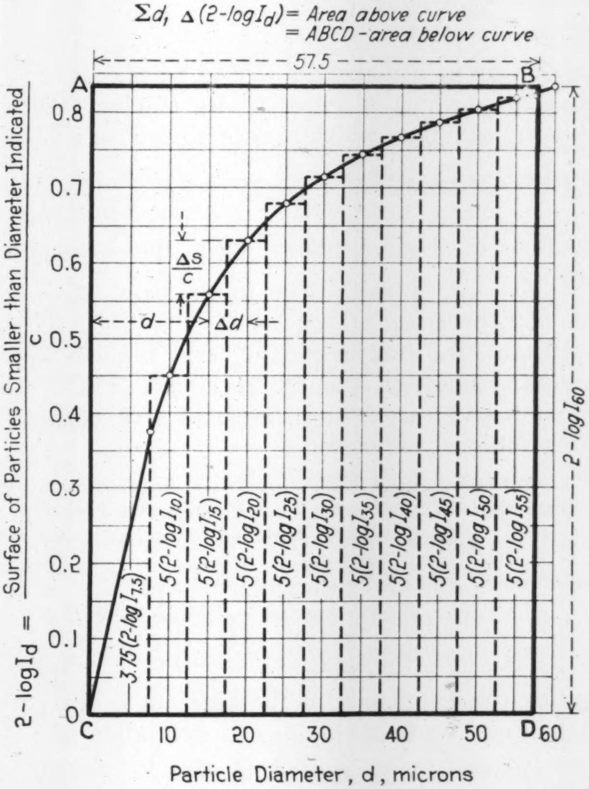

FIG. 3.—Typical Particle Size - Surface Area Distribution Curve for Portland Cement.

By taking a small size increment of the distribution curve of a sample of cement (Fig. 3) of limiting sizes $d$ and $d + \Delta d$, the average diameter, $d_1$, of all the particles in the increment may be taken as the average of the limiting sizes or $d_1 = d + \frac{\Delta d}{2}$. The surface area, $\Delta S$, of the particles in the same increment is obtained from Eq. 2 as follows:

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[1] The specific gravity of portland cement does not vary greatly and in this work it is considered constant at 3.15. A variation of 0.15 from this value when substituted in Stokes' law gives a variation of 2.5 per cent in the diameter of the particle measured.

$$\begin{aligned}\triangle S &= S_{d+\Delta d} - S_d\\ &= c\,[(\log I_o - \log I_{d+\Delta d}) - (\log I_o - \log I_d)]\\ &= c(\log I_d - \log I_{d+\Delta d})\end{aligned}$$

By substituting this value of $\triangle S$ in Eq. 3, the weight, $\triangle w$, of the particles in the increment is expressed as follows:

$$\triangle w = 0.0000525\, d_1\, c(\log I_d - \log I_{d+\Delta d}) \qquad (4)$$

Although the values of $\triangle w$ are not known, since $c$ is still unknown, their summation up to a maximum size of 60 microns is the weight of the sample, $W_{60}$, which is known.[1] By making a summation of $\triangle w$ the following equation, which may be solved for $c$, is obtained:

$$\sum_{d=0}^{d=60} \triangle w = W_{60} = 0.0000525\, c \sum_{d=0}^{d=60} d_1 (\log I_d - \log I_{d+\Delta d}) \qquad (5)$$

An examination of Fig. 3 shows that the summation, $\sum_{d=0}^{d=60} d_1 (\log I_d - \log I_{d+\Delta d})$, is equivalent to the area above the curve. With values of $I_d$ observed at 7.5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55 and 60 microns, the area below the curve is easily approximated and when subtracted from the full rectangle, *ABCD*, the desired area above the curve (neglecting the small area between 57.5 and 60 microns) is obtained as follows:

Area of the full rectangle, $ABCD = 57.5\,(2 - \log I_{60})$.
Area under curve $= 3.75\,(2 - \log I_{7.5}) + 5\,(2 - \log I_{10}) + 5\,(2 - \log I_{15}) + 5\,(2 - \log I_{20}) \ldots + 5\,(2 - \log I_{55})$.
Area above curve = area full rectangle minus area below curve $= 57.5\,(2 - \log I_{60}) - 3.75\,(2 - \log I_{7.5}) - 5\,(2 - \log I_{10}) - 5\,(2 - \log I_{15}) - 5\,(2 - \log I_{20}) \ldots - 5\,(2 - \log I_{55})$.

By substituting this expression of the area above the curve for the summation in Eq. 5 and simplifying, an evaluation of $c$ is obtained in which the quantities are known or can be observed with the turbidimeter:

$$c = \frac{3810\, W_{60}}{1.5 + 0.75 \log I_{7.5} + \log I_{10} + \log I_{15} + \log I_{20} \ldots + \log I_{55} - 11.5 \log I_{60}} \qquad (6)$$

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[1] The curve is not obtained for particles larger than 60 microns because the larger particles are relatively few in number and have such a small surface in proportion to their weight that changes in turbidity caused by their falling from the suspension are not indicated reliably. Since the No. 325 sieve passes an average maximum particle of about 60 microns, the weight of that part of the sample finer than 60 microns is obtained from a No. 325 sieve determination made on a separate sample of the cement. Assuming the No. 325 sieve separation to be at 60 microns,

$$W_{60} = \frac{\text{weight of sample} \times \text{per cent passing No. 325 sieve}}{100}$$

Using this value of $c$, Eq. 2 may be solved for $S_{60}$ as follows:

$$S_{60} = \frac{3810\, W_{60}\, (2 - \log I_{60})}{1.5 + 0.75 \log I_{7.5} + \log I_{10} + \log I_{15} + \log I_{20} \ldots\ldots + \log I_{55} - 11.5 \log I_{60}} \quad (7)$$

where $S_{60}$ and $W_{60}$ are the surface and weight of the particles less than 60 microns in diameter.

To evaluate $S_{60}$ in terms of a 1-g. sample, that is, to arrive at the specific surface of the sample, it is necessary to divide the value obtained for $S_{60}$ by the weight of the sample used. This cancels with the weight of sample used in determining $W_{60}$ and it is seen that the actual weight of the sample drops out of the calculation.

Neglecting the surface area of the particles larger than 60 microns and dropping the decimal from the constant 38.1, the specific surface of any sample then becomes:

$$S = \frac{38r\, (2 - \log I_{60})}{1.5 + 0.75 \log I_{7.5} + \log I_{10} + \log I_{15} + \log I_{20} \ldots\ldots + \log I_{55} - 11.5 \log I_{60}} \quad ..(8)$$

where $S$ = specific surface of the sample in square centimeters per gram,
$r$ = percentage by weight passing a No. 325 sieve,
$I_{7.5}, I_{10}, I_{15}, I_{20} \ldots\ldots$ = intensity of light, in microamperes, transmitted through a suspension from which all particles larger than 7.5, 10, 15, 20, . . . . have settled past the level of the light beam.

If the size-weight distribution of the sample is desired, it is obtained by first determining $c$ (Eq. 6) and then substituting the value of $c$ thus obtained in Eq. 4 and solving for the increments of $\triangle w$.

## APPLICATIONS AND LIMITATIONS

In the use of Eq. 1, Stokes' law, for selecting the time intervals, $t$, and depths of suspension, $h$, at which the microammeter should be read to correspond to particle diameters of 7.5, 10, 15, 20, . . . 60 microns, the density and absolute viscosity of the dispersing medium must be known. Since not all laboratories are equipped to determine these properties of the dispersing medium, the Bureau of Standards has prepared a standard cement sample by use of which the constants of the dispersing medium are derived with the apparatus itself. This is accomplished by observing the settling time of the standard sample in the kerosine to be calibrated and comparing this observed time with the settling time which has been previously established for the same sample in a kerosine of known viscosity and density. The use of the standard sample for calibration of the apparatus is described in detail in the Appendix.[1]

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[1] See p. 566.

*Correction for Temperature.*—Since the viscosity of the dispersing medium changes with temperature, it is necessary either that the apparatus be operated at the temperature for which it is calibrated or that corrections be made for changes in temperature. The latter is conveniently accomplished by a timing burette which is filled with kerosine at the same temperature as that of the standard suspension. Flow of kerosine from the burette and the settling of the particles from the suspension are started at the same time. The rate at which the kerosine drains from the burette varies with temperature in very nearly the same ratio as the rate of fall of the particles in the suspension. The levels to which the kerosine drains in the calculated time intervals at which microammeter readings are to be made are marked on the burette. After the burette has been graduated at a known temperature it may be used as a timer for reading the microammeter within the normal range of room temperature without further correction. Details for calibration and use of the burette are given in the Appendix.[1]

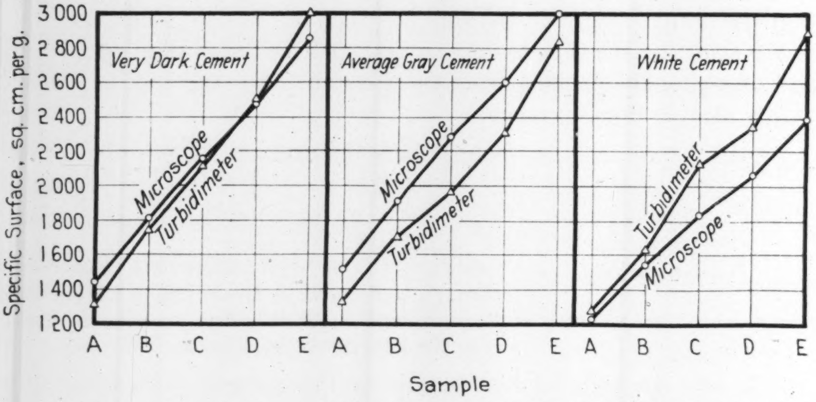

FIG. 4.—Comparison of Values of Specific Surface Obtained with the Turbidimeter and with the Microscope.

NOTE.—Samples were blended from fractions of ground clinker as shown below:

| | Per Cent by Weight of Each Fraction | | | | |
|---|---|---|---|---|---|
| Nominal size of fraction, microns | 0 to 7 | 7 to 22 | 22 to 45 | 45 to 75 | >75 |
| Sample A | 15 | 15 | 15 | 25 | 30 |
| Sample B | 20 | 20 | 20 | 20 | 20 |
| Sample C | 25 | 25 | 20 | 15 | 15 |
| Sample D | 30 | 25 | 20 | 15 | 10 |
| Sample E | 35 | 30 | 25 | 10 | 0 |

*Adaptability to Routine Testing.*—For cement produced at a single plant it is probable that $c$ will not vary significantly and for

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[1] See p. 566.

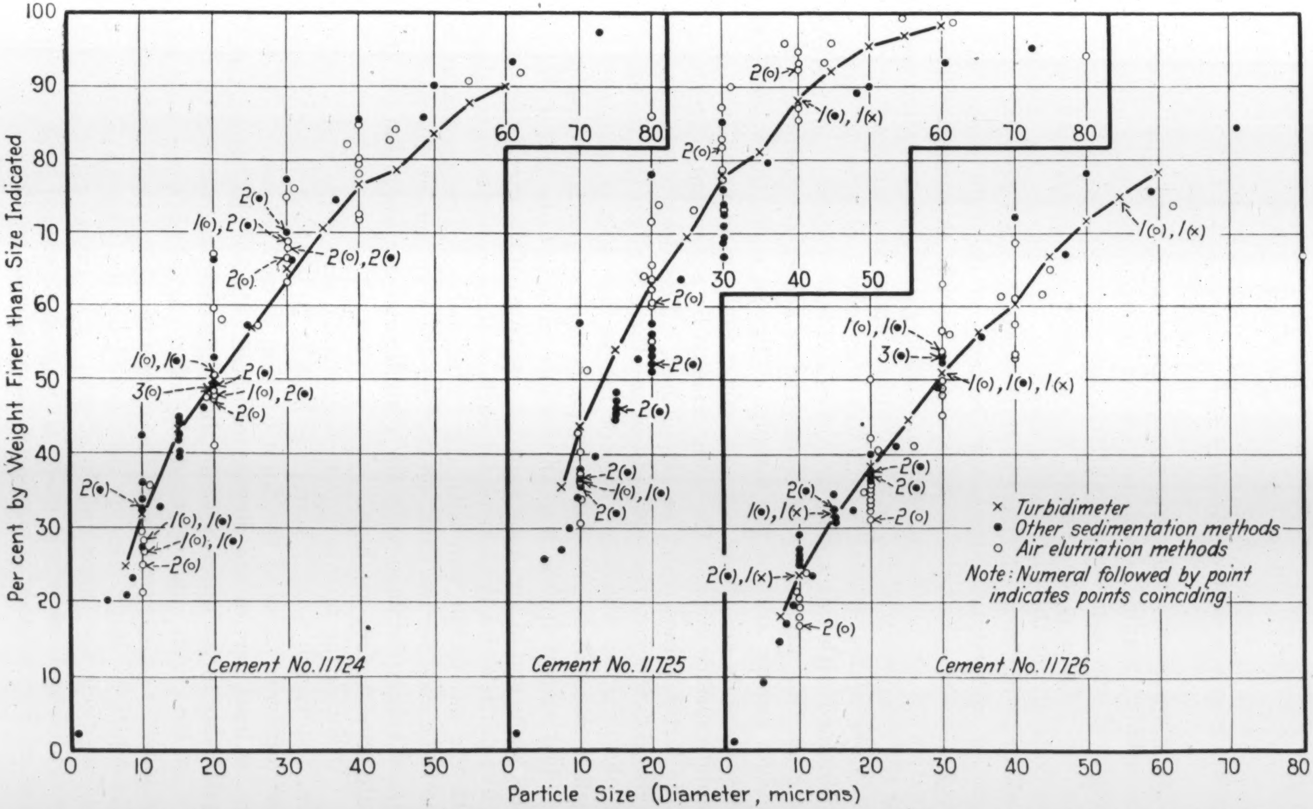

FIG. 5.—Size-Weight Distribution of Three Portland Cements as Determined with the Turbidimeter and by Other Laboratories Using Different Methods.

routine determinations on such a product the specific surface can be readily approximated from Eq. 2 using the ratio

$$\frac{\underline{S}\text{ (unknown sample)}}{\underline{S}\text{ (known sample)}} = \frac{2 - \log I_{60}\text{ (unknown sample)}}{2 - \log I_{60}\text{ (known sample)}}$$

The operation of the apparatus then becomes very rapid as only one turbidity reading is required for the determination of the specific surface of the sample. Additional readings are needed only to establish *c* for a given cement, or to obtain size distribution data. Readings giving the distribution down to 7.5 microns are obtained in about 10 minutes, which is the time required for all particles larger than 7.5 microns to settle below the level of the light beam, with this particular apparatus and suspending liquid. A complete determination on an unknown sample, including the determination of *c* and the calculation of the results, requires about one-half hour.

*Scope of the Method.*—Although the values of specific surface as obtained with this apparatus are calculated as square centimeters per gram they are not to be considered as accurately describing the absolute surface area of the sample. That is, while the values are fairly accurate relatively, all the values may be in error from the absolute surface areas of the same cements by a constant percentage, if the latter values could be established. This does not indicate a serious deficiency in the method because there is at present no means for obtaining an absolute value of the surface area of particles as small in size and as irregular in shape as those of portland cement.

The nearest approach to an absolute value is to be had with the microscope, and in Fig. 4 a comparison is shown of the specific surfaces of five particle-size gradations for each of three cements as obtained with the microscope and with the turbidimeter. The agreement is seen to be good for the dark cement; for the gray cement, the specific surface values obtained with the turbidimeter are low; and for the white cement, they are high.

Although the microscopic measurements were made with utmost care, a spread in specific surface as high as 20 per cent was obtained from duplicate slides of the finest fraction (0 to 7 microns). Therefore, at least part of the divergence noted between the two methods may be due to the uncertainty of the microscopic measurements.

In Fig. 5 the size-weight distribution curves obtained with the turbidimeter are compared with those determined by other laboratories on samples of three cements distributed by the Cement Reference Laboratory at the Bureau of Standards. The turbidimeter analyses (computed on a size-weight basis) are in good agreement with analyses obtained by the other laboratories using different methods.

## TURBIDIMETER ANALYSES OF TWENTY CEMENTS

In Table I are given the results of duplicate determinations of the specific surface of 20 portland cements. The values of specific surface were calculated from Eq. 8.

The values of the "transmittancy constant," *c*, (Table I) range from 665 for a very dark cement to 985 for a white cement.

Duplicate analyses on the same cement, except for cement No. 2, do not differ more than 5 per cent. In general, an agreement within 5 per cent may be expected unless the cement is lumpy or partially hydrated and a complete dispersion of the sample is not attained.

TABLE I.—SPECIFIC SURFACE OF TWENTY PORTLAND CEMENTS.

| CEMENT | ANALYSIS NO. 1, SQ. CM. PER G. | ANALYSIS NO. 2, SQ. CM. PER G. | AVERAGE TRANSMITTANCY CONSTANT, *c* |
|---|---|---|---|
| No. 1 | 1090 | 1090 | 735 |
| No. 2 | 1150 | 1220 | 725 |
| No. 3 | 1410 | 1390 | 720 |
| No. 4 | 1470 | 1400 | 710 |
| No. 5 | 1480 | 1500 | 760 |
| No. 6 | 1580 | 1510 | 735 |
| No. 7 | 1580 | 1550 | 750 |
| No. 8 | 1690 | 1610 | 690 |
| No. 9 | 1680 | 1640 | 755 |
| No. 10 | 1750 | 1690 | 745 |
| No. 11 | 1710 | 1700 | 690 |
| No. 12 | 1760 | 1710 | 745 |
| No. 13 | 1880 | 1810 | 665 |
| No. 14 | 1870 | 1910 | 805 |
| No. 15 | 1950 | 1920 | 790 |
| No. 16 | 1990 | 1910 | 720 |
| No. 17 | 1950 | 1980 | 690 |
| No. 18 | 2120 | 2120 | 690 |
| No. 19 | 2200 | 2190 | 985 |
| No. 20 | 2440 | 2490 | 810 |

*Acknowledgments:*

The author desires to acknowledge the generous response of the many members of the staff of the Bureau of Standards whose advice and assistance have greatly facilitated this investigation.

Especial acknowledgment is due P. H. Bates who suggested the general outline of the work and whose continued interest and counsel have been of material assistance in the development of the apparatus and of the method for its use.

## APPENDIX

### DETAILS OF CONSTRUCTION AND OPERATION OF THE TURBIDIMETER

*Apparatus Required:*

1. *Stirring Motor.*—A brush which is clipped to fit the contour of the bottom of a test tube is attached to a stirring motor. The brush should rotate about 500 r.p.m.

2. *Battery.*—6-v. automobile storage battery.

3. *Timing Burette.*—The timing burette consists of a glass tube of about 18 mm. inside diameter and about a meter in length. A capillary about a millimeter in diameter and of sufficient length to drain the burette in about 15 minutes is fastened into the bottom of the larger tube by means of a cork stopper. A line, called the zero line, should be marked or etched on the burette about 15 cm. from the top. The burette is conveniently supported by means of two burette clamps fastened to a 36-in. ring stand.

4. *Microammeter.*—A microammeter of 0 to 50 microamperes range is used. The internal resistance of the microammeter should not be greater than 100 ohms.

5. *Suspending Liquid.*—At least 20 gal. of any clear colorless kerosine should be set aside for use with this apparatus. The used kerosine is easily reclaimed by allowing the cement to settle and siphoning off the clear liquid. If it becomes colored it can be decolorized by stirring some filter charcoal in with it before settling. Reclaiming the kerosine in this manner does not change its viscosity or density, and recalibration for its reuse is not required.

6. *Turbidimeter.*—The turbidimeter proper, with the more important dimensions, is shown in Fig. 1.

The source of light, *L*, is a 6-candle-power electric lamp operated by a storage battery. A parabolic reflector is mounted behind the lamp and is focused to throw a beam of approximately parallel light through the settling tank, *T*, and into the photo-electric cell, *P*. The light intensity is adjusted by means of 6 and 30-ohm rheostats, $R_1$ and $R_2$, mounted in series with the lamp but in parallel with each other.

A water cell, *C*, is placed in the path of the light to absorb the larger part of the radiant heat in the beam. The cell used consists of a piece of 3-in. brass tubing 4 in. long with glass windows cemented with ordinary sealing wax into the ends of the tube. A stoppered hole in the top of the cell provides for filling with distilled water.

The settling tank is made of $\frac{3}{16}$-in. plate glass cemented together to form a rectangular tank of the following inside dimensions: cross-section, 2 by $1\frac{1}{2}$ in.; height, 8 in. The 2-in. faces of the tank are placed perpendicular to the beam of light. The two 2-in. faces should be equidistant within 0.003 in. at all points. A mark is placed on the side of the tank to indicate a volumetric content of 335 ml.

A Weston Photronic photo-electric cell is used to measure the intensity of the light which is transmitted through the tank. The photo-electric cell is connected directly to the microammeter. A hood carying a horizontal slot $\frac{1}{2}$ by $1\frac{3}{8}$ in. is mounted over the face of the photo-electric cell.

7. *Retarding Filter.*—An Eastman Filter No. 35D or equivalent. Any colored glass which will reduce the intensity of the light from a reading of 100 microamperes to a reading of 20 to 30 amperes is satisfactory.

8. *Mounting.*—The source of light, water cell and photo-electric cell are mounted on a movable shelf which is raised or lowered by two lead screws connected by a chain. The settling tank is mounted on a base which is independent of the cabinet and of the rest of the apparatus so that the tank will be as free as possible from vibration.

The level of the light beam with reference to the surface of the suspension is indicated by a pointer which travels along a scale mounted on the right-hand end of the cabinet, as shown in Fig. 1. In setting up the apparatus the height of the base of the tank should be adjusted so that the *surface of the liquid in the tank (when containing 335 ml.) is at the same level as the center line of the slots for the light beam when the shelf pointer is at the zero mark on the scale.* The lines on the scale marked 7.5, 10, 15, 20, 25, and 30–60 indicate the positions at which the pointer should be located when turbidity readings for these particle sizes are taken. The lines are located on the scale at distances from the zero mark equal to the values of *h* in Table II.

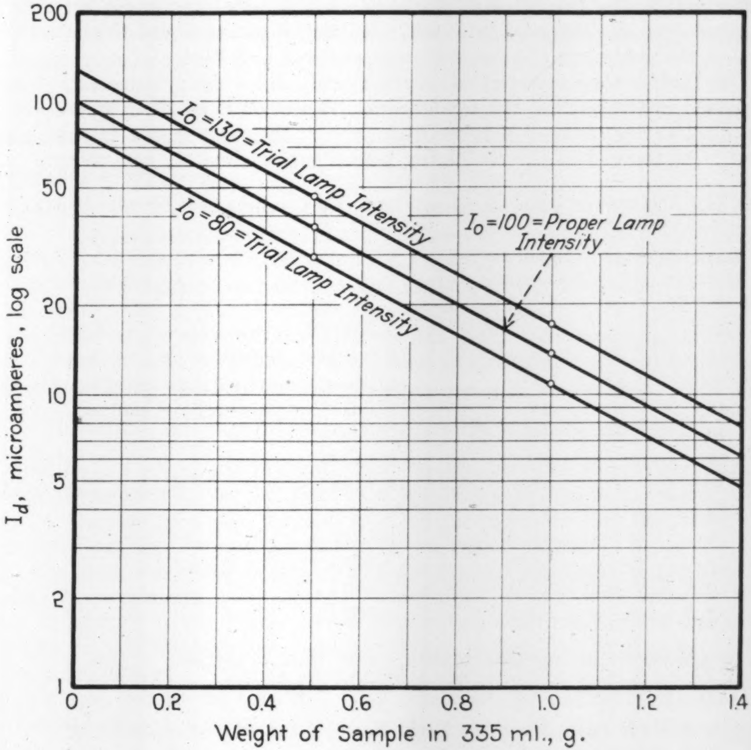

FIG. 6.—Illustrating Method of Determining the Value $I_0$ from Turbidity Readings of Two Concentrations of a Suspension.

9. *U. S. Standard No. 325 Sieve.*—A sieve, 2 or 3 in. in diameter and having walls 3 or 4 in. high, is convenient. For washing the sample through the sieve, a spray nozzle having seventeen 0.02-in. holes in the tip should be used. A

pressure gage should be installed in the water line between the shut-off valve and the nozzle so that the pressure on the nozzle can be regulated.

*Dispersion of the Sample:*

The sample is placed in a test tube with 15 ml. of kerosine and 5 drops of oleic acid. This mixture is stirred for one minute with the rotating brush and is then transferred to the tank. The test tube is washed clean with fresh kerosine and finally the brush is washed in a test tube containing kerosine to which one drop of oleic acid is added. All washings are added to the settling tank and the volume of the suspension is adjusted to 335 ml.

The tank is then agitated to obtain a uniformity of the suspension before it is placed in the path of the light beam. Agitation is effected by oscillating the tank 180 deg. around a horizontal axis at the rate of one inversion per second. At the end of the oscillating period (approximately one minute) the tank is placed in an upright position, and if any kerosine drains down the outside of the tank it should be immediately wiped away.

TABLE II.—VALUES OF $h$ AND $d$ TO BE USED FOR CALIBRATION OF THE APPARATUS.

| PARTICLE DIAMETER, $d$, MICRONS | DEPTH OF SUSPENSION, $h$, CM. | $\frac{h}{d^2}$ | SETTLING PERIOD, $T$, MINUTES $\left(=\frac{th}{d^2}\right)$ |
|---|---|---|---|
| 60 | 15 | 0.00417 | This column is to be filled in by operator after the interval, $t$, has been determined. |
| 55 | 15 | 0.00496 | |
| 50 | 15 | 0.00600 | |
| 45 | 15 | 0.00741 | |
| 40 | 15 | 0.00938 | |
| 35 | 15 | 0.01225 | |
| 30 | 15 | 0.01667 | |
| 25 | 13.1 | 0.0210 | |
| 20 | 10.0 | 0.0250 | |
| 15 | 6.6 | 0.0293 | |
| 10 | 3.3 | 0.0333 | |
| 7.5 | 2.1 | 0.0375 | |

*Adjustment of the Intensity of the Light Beam, $I_0$, to 100 Microamperes:*

A suspension containing 0.5 g. of the standard sample is dispersed as described. The tank is immediately placed in the apparatus, and with the scale pointer at the 30–60 position, a microammeter reading is noted at exactly one minute after the agitation of the tank was stopped. Following the same procedure a second suspension is prepared using 1 g. of the same cement and a second microammeter reading is obtained. These two readings are plotted as ordinates on semilogarithmic paper against the weight of sample, as shown in Fig. 6. A straight line is drawn through the two points and the intersection of this line with the axis of zero weight of sample gives the intensity of the beam, or $I_0$. If the value of $I_0$ thus obtained is other than 100 microamperes, the intensity of the lamp is readjusted and the process repeated until this value is obtained.

When the lamp has been adjusted to the desired intensity, the filter is placed in the path of the light beam and the tank is removed. The reading with the filter in place then becomes a permanent reference value, $I_0 \times f$, for the intensity of the light and the lamp must be adjusted to give this value before each analysis. If the lamp, photo-electric cell, or any other part of the optical system is changed, the value of the intensity of the beam through the filter must be redetermined. A value of $I_0$ of about twice the range of the microammeter is arbitrarily chosen so that when a cement suspension is placed in the path of the beam, the full range of the microammeter is useful for indicating turbidity. If a lower intensity within the range of the microammeter be chosen for $I_0$, the intensity of the light transmitted through the suspension will not be sufficient to use more than the lower fourth of the microammeter scale. It is desirable to have a reference value of $I_0$ of reduced intensity. The photo-electric cell has a drift or fatigue characteristic, and by using a reference value $I_0 \times f$, which is about the same intensity as the light transmitted through the suspension, the error due to drift of the photo-electric cell is largely avoided.

*Calibration of the Burette:*

A scale for the burette, showing the levels reached by the kerosine at the times when microammeter readings should be taken, is prepared in the following manner. A suspension containing 1 g. of the standard sample is prepared and after agitating for one minute, the tank is placed in position in the path of the light beam at the 15-cm. depth, having adjusted the lamp as previously described. A timing clock is started at the instant oscillating of the tank is stopped and the elapsed time, $t$, in seconds is determined at which the microammeter reading specified for the standard sample is obtained. The values of $\frac{h}{d^2}$ in Table II are multiplied by $t$ to give the time intervals, $T$, $\left(T = \frac{th}{d^2}\right)$ in minutes, at which the microammeter should be read to give the turbidity values corresponding to the particle sizes shown in the first column of the table. The depths of the suspension at which the shelf must be placed when these readings are obtained are shown in the second column.

The time intervals, $T$, are valid only for the temperature at which the calibration was made. The burette should next be filled with kerosine at this same temperature, the timing clock should then be started at the instant the kerosine reaches the zero level, and the levels reached by the draining kerosine in the time intervals $T$ should be marked on the burette or on a strip of heavy paper placed behind the burette. The corresponding particle diameters shown in Table I should also be inscribed at these levels. This completes the calibration and the apparatus may be used within the normal range of room temperatures without further correction, the change in the rate of flow of the kerosine from the burette automatically compensating for change in viscosity of the suspension due to temperature.

*Separation with the No. 325 Sieve:*

A 1-g. sample is washed by placing the sieve under the spray nozzle for one minute, the pressure on the nozzle having been previously adjusted to 10 lb. per sq. in. After washing is completed the sieve and residue are dried in an oven or on a hot plate at a temperature not exceeding 120 C., after which the residue is brushed from the sieve and weighed. After having been used for 10 or 15 determinations, the sieve should be dipped in a 10 per cent solution of hydrochloric acid and immediately washed with water. This treatment does not injure the sieve and serves to remove cement particles which have become lodged in the meshes.

*Size of Test Sample:*

The size of sample for the turbidimeter is selected so that the microammeter readings will fall in the middle portion of the scale. A sample of 0.3 g. for a very fine cement, 0.4 g. for a cement of medium fineness, and 0.5 g. for a coarse cement has been found satisfactory. A 1.0-g. sample is used for determinations of residue on the No. 325 sieve.

*Test Procedure:*

1. The sample is dispersed and placed in the settling tank, clear kerosine is added to bring the volume to the 335-ml. mark, and the windows of the tank are wiped clean with a piece of soft cloth.

2. The intensity of the lamp is adjusted with the filter in place to the proper value, $I_o \times f$, the filter being left in the path of the beam until the suspension has been placed in position.

3. The shelf pointer is placed at the 30–60 mark on the scale.

4. The burette is filled with kerosine.

5. Oscillation of the tank is commenced immediately and is continued until the kerosine drains to the zero level in the burette, at which time the tank is immediately placed in position in the path of the light beam.

6. The filter is removed from the path of the light beam and the door of the cabinet is closed.

7. With the shelf pointer at the 30–60 mark, microammeter readings are taken at the instants the kerosine in the burette drains past the marks 60, 55, 50, 45, 40, 35 and 30. The shelf must then be raised to the corresponding levels indicated by the pointer and scale before each of the succeeding readings, which are taken at the instants the kerosine drains past the 25, 20, 15, 10, and 7.5 marks on the burette.

8. At the end of the test the filter is placed back in the path of the light beam, the tank is removed,[1] and another reading of $I_o \times f$ is obtained. If a change in the intensity of the light of more than 0.2 microampere has taken place the test should be repeated.[2]

9. A No. 325 sieve determination is made on another sample of the same cement, the percentage passing being denoted as $r$.

10. The specific surface of the sample is calculated from the equation:

$$\underline{S} = \frac{38r\ (2 - \log I_{60})}{1.5 + 0.75\ \log I_{7.5} + \log\ I_{10} + \log\ I_{15} + \log I_{20}\ .\ .\ .\ + \log I_{55} - 11.5 \log I_{60}}$$

where $\underline{S}$ = specific surface of sample in square centimeters per gram,

$r$ = per cent by weight of sample passing a No. 325 sieve, and

$I_{7.5}$, $I_{10}$, $I_{15}$, etc. = microammeter readings which correspond to particle sizes of 7.5, 10, 15, etc., microns (Table II).

Having determined the specific surface of a sample of cement from a given plant, the specific surface of subsequent samples from the same plant can be quickly obtained by observing only the first turbidity reading, $I_{60}$, of the subsequent suspensions and calculating their specific surface from the ratio:

$$\frac{\underline{S}\text{ (unknown sample)}}{\underline{S}\text{ (known sample)}} = \frac{2 - \log I_{60}\text{ (unknown sample)}}{2 - \log I_{60}\text{ (known sample)}}$$

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[1] Either the filter or the tank containing a suspension of cement or both must be in the path of the light beam at all times. If not, the full strength of the light beam will drive the pointer off the microammeter scale and possibly injure it. *This is important.*

[2] If a freshly charged storage battery be short-circuited momentarily before it is used, its voltage will remain very constant until it is nearly discharged.

# CALORIMETER INSTALLATION IN THE ENGINEERING MATERIALS LABORATORY OF THE UNIVERSITY OF CALIFORNIA FOR STUDIES OF HEAT GENERATION IN MASS CONCRETE

By S. B. Biddle, Jr.,[1] and J. W. Kelly[1]

## Synopsis

Outstanding features of the cement investigations for the Boulder Dam are the measurement of the heat generated by 93 cements during hydration and the curing of specimens for strength and volume-change tests under variable temperature conditions corresponding to those in mass concrete. The system of calorimeters and curing chambers employed at the University of California is described in this paper.

Direct measurements of temperature rise in mass concrete are made for certain cements in six adiabatic calorimeters, and indirect measurements of heat generation of cement are made for all cements in a heat-of-solution calorimeter by dissolving neat-cement samples in acid. Approximate curing temperatures corresponding to those of mass concrete are determined in advance by measuring temperature rise of concrete specimens in high-insulation calorimeters. The curing is then carried out in variable-temperature chests, controlled by specially designed instruments.

The comparative advantages of the different methods of determining heat generation are herein pointed out, and the function of each method in the coordinated program of cement investigation is shown.

## Introduction

In mass-concrete construction, the problem of heat evolution by the hydrating cement is of primary importance, not only because of thermal expansion of the mass but also because of the effect of variations in temperature conditions during hydration upon the strength and other related properties of the concrete.

Determinations of the rate and total quantity of heat generated by 93 commercial and laboratory cements of widely varying characteristics have been made in the Engineering Materials Laboratory of the University of California, as part of the general cement investigations undertaken for Boulder Dam by the U. S. Bureau of Reclamation. In this paper are described the methods and apparatus used.

Two methods of determining heat generation of cement are employed. The first, which, because of its relatively high cost of operation is applied to only a few cements, measures the temperature rise during hydration of large concrete specimens cured in adiabatic calorimeters. The second, an indirect method which is applied to all of the cements under investigation, measures the change in heat energy of neat-cement samples through determinations of their heat of solution at various ages. The basic values determined for the selected cements by the temperature-rise method serve as a control and check on the indirect method.

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## ADIABATIC CALORIMETERS

For the few selected cements, determinations of heat generation by the temperature-rise method are being carried on in a set of six adiabatic calorimeters built in this laboratory. The construction details of these calorimeters have been changed with the development of the method, but all are essentially as shown in Fig. 1. Each consists of an insulated room in which the air temperature is accurately controlled in a range extending up to about 200 F. (95 C.). The shape of the room is such that fans suitably placed keep the air in continuous circulation and hence at a uniform temperature throughout, even when the temperature is being changed at a rate of several degrees per hour. As the heating units are mounted directly on the fans, the heat released in the room is rapidly distributed and temperature inequalities do not develop.

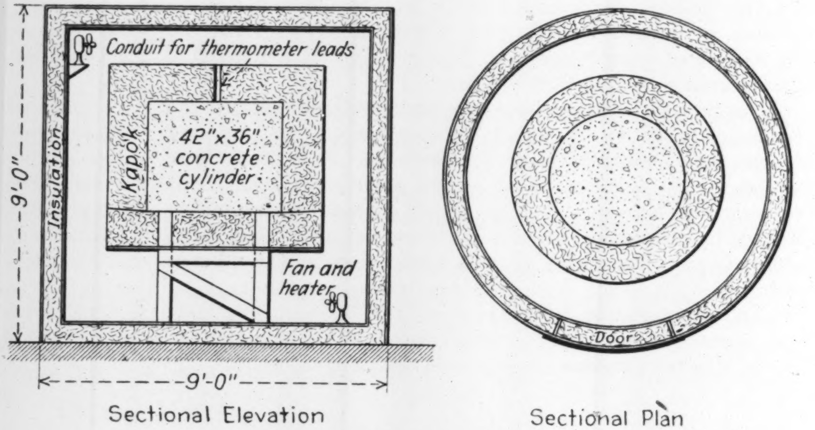

FIG. 1.—Adiabatic Calorimeter.

In order that the concrete in the specimens may be closely similar to that planned for Boulder Dam, cylindrical specimens ranging in size up to 42 by 36 in. are used. In these large specimens, the consistency and water-cement ratio of the mix, the cement content, the gradation of the aggregate, and the aggregate itself (9-in. maximum size) are precisely as proposed for Boulder Dam.

Each test specimen is placed in a light metal container and sealed to prevent loss of moisture. The specimen is placed in an adiabatic calorimeter room as shown in Fig. 1, and the temperature of the air in the calorimeter is controlled to be the same at all times as the temperature of the specimen. As there is no appreciable thermal head under these circumstances, the flow of heat is negligible and the specimen rises in temperature as a result of the heat generation of the cement which it contains. The exactitude with which this "zero" thermal head is maintained governs to a large extent the accuracy of the test, particularly if the test is carried on for an extended period. Inasmuch as a small temperature difference must exist before a change is made in the air temperature, the temperature of the air is controlled in a series of short steps and is alternately higher and lower than the temperature of the specimen. The effect of these minute temperature differences is rendered negligible by a shield of kapok insulation which surrounds the specimen. The heat capacity of the container and of this insulation is compensated by the use of a calculated amount of additional cement in the specimen.

*Automatic Control:*

Extreme care has been taken in the construction and operation of the two adiabatic calorimeters in which long-time tests are made. The air temperature is controlled by automatic temperature-difference controllers. Each of these controllers is electrically a Wheatstone bridge, of which two arms are fixed resistances and the other two are sensitive copper resistance thermometers. One of these thermometers is placed in the concrete specimen and the other in the air of the calorimeter. When the temperature of the specimen rises above that of the calorimeter air, the controller closes a relay, and additional heat is released in the air until the temperatures are once more equal. As the instruments are of the recording type, the extent and duration of any temperature difference is readily determined, and any accumulated difference can be compensated by an adjustment of the instrument. The sensitivity of each instrument is such that it will detect a differential temperature of the order of 0.01 F. (0.006 C.) It is possible to operate one of these calorimeters for a period of several months with a temperature difference which seldom exceeds 0.1 F. (0.06 C.), and with an accumulative average difference close to zero.

Two additional resistance thermometers placed in the concrete permit determination of the temperature rise of the specimen to the nearest 0.1 F. (0.06 C.) by means of auxiliary measuring equipment. Additional thermometers are also placed in the calorimeter air to provide a check on the performance of the automatic controller.

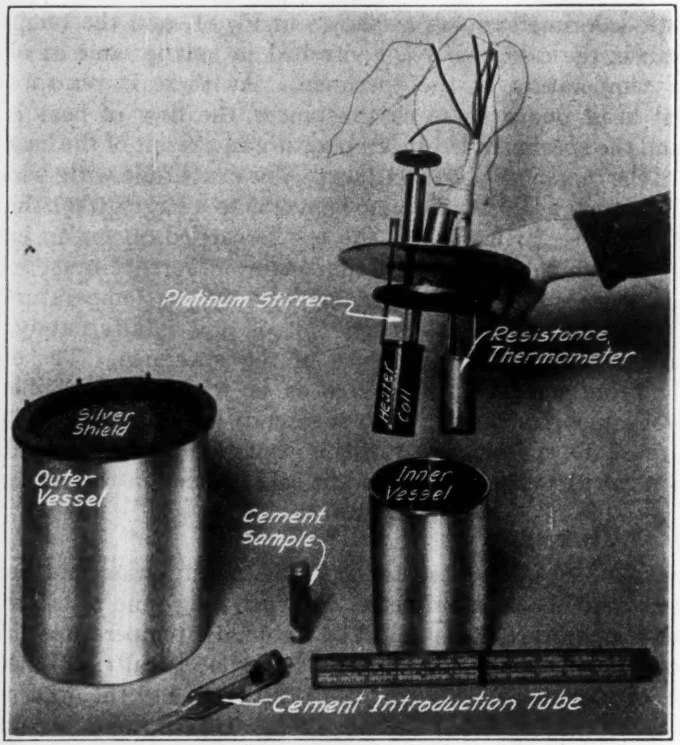
Platinum Stirrer
Resistance Thermometer
Heater Coil
Silver Shield
Outer Vessel
Inner Vessel
Cement Sample
Cement Introduction Tube

FIG. 2.—Heat-of-Solution Calorimeter.

*Manual Control:*

The remaining four calorimeters, which are designed for short-time tests of a month or less, are closely similar in construction to the automatic type, but the air temperature is controlled manually. The temperatures of the calorimeter air and the concrete test specimen are measured independently, and a helical bimetallic thermostat which is sensitive and adjustable to a few tenths of a degree is set to maintain the air temperature at the correct value. This adjustment is made at sufficiently frequent intervals to insure an average thermal head of less than 1 F. (0.6 C.). After the first day or so of a test the temperature of the test specimen changes only a few degrees per day, and the thermal head can be kept less than 0.5 F. (0.3 C.).

## HEAT-OF-SOLUTION METHOD

Even if it were considered desirable to do so, a very large amount of space, equipment, and material would be required to make an extended heat-generation study of 93 different cements in adiabatic calorimeters. To make this major study, the heat-of-solution method, which was first applied to portland cement by the technical department of the Riverside Cement Co.,[1] has been further developed in this laboratory. Although the original cost of heat-of-solution equipment is several times that of an adiabatic calorimeter, the heat-of-solution method is more economical for cement acceptance tests or for any program involving a large number of samples. It is rapidly becoming

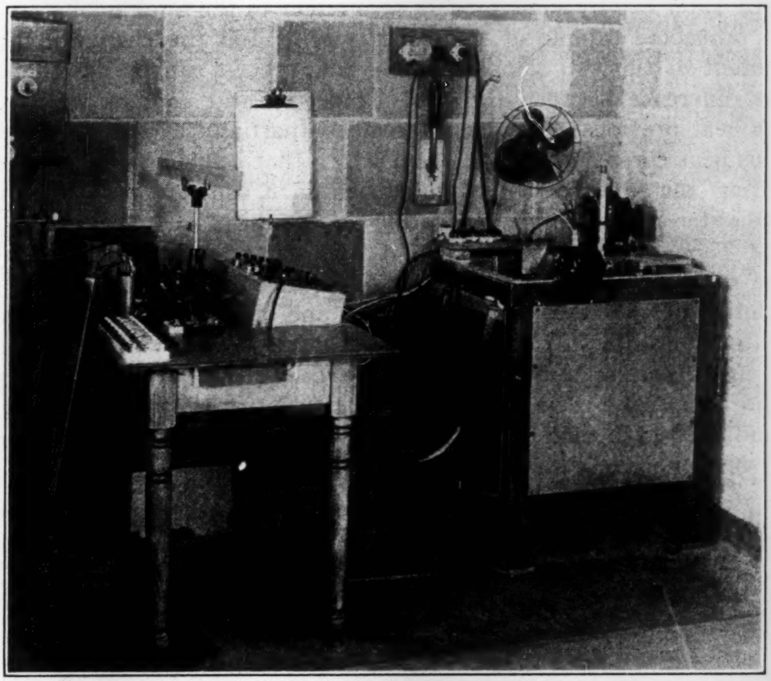

FIG. 3.—Bath and Auxiliary Measuring Equipment for Heat-of-Solution Calorimeter.

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[1] Hubert Woods, H. H. Steinour, and H. R. Starke, "Effect of Composition of Portland Cement on Heat Evolved During Hardening," *Industrial and Engineering Chemistry*, Vol. 24, p. 1207, November, 1932.

a generally accepted method, and has been specified (1932) for cement acceptance tests for the Pine Canyon Dam of the City of Pasadena, and more recently for Boulder Dam.

Hess' Law of Constant Heat Summation states that the change in the heat content of a system in passing from one state to another is independent of the path whereby the passage is made. If, therefore, a cement which has previously lost heat during a period of reaction with water can by some process be brought to the same final state as an unreacted sample of the same cement, and the change in heat content of the two samples during this process can be determined, the difference between the two changes in heat content will equal the heat previously lost by the reacted (partially hydrated) sample. This heat corresponds to the heat which that cement would generate during the same period of hydration in a concrete mix, provided the water-cement ratios and curing temperatures were the same.

These changes in heat content, and hence the heat generation of the cement, are determined in the heat-of-solution method by the solution of unreacted cement samples, and of corresponding partially hydrated samples at various ages of hydration, in a dilute mixture of nitric and hydrofluoric acids in a calorimeter of special design. The equipment is so designed, and the entire testing procedure is conducted with such accuracy, that duplicate determinations of heat of solution agree within 0.3 per cent.

### *Heat-of-Solution Calorimeter:*

A photographic view of the heat-of-solution calorimeter is shown in Fig. 2, and a view of the water bath and auxiliary measuring equipment is given in Fig. 3. The essential parts are as follows:

1. *Inner Vessel.*—Capacity, 1200 ml. Its walls and bottom are of $\frac{1}{64}$-in. copper, with an inner sheath of pure gold $\frac{1}{64}$ in. thick. It is chromium-plated and polished on the outside.

2. *Cover for Inner Vessel.*—Copper, chromium-plated and polished on the outside, and tight-fitting and removable. The inner surface of the cover is protected against acid attack by a coating of beeswax. The cover serves as a support for the stationary parts which are in the inner vessel during test, and provides an effective evaporation seal around all moving parts which enter this vessel.

3. *Outer Vessel.*—$\frac{3}{32}$-in. brass, chromium-plated and polished inside and outside, and of a size sufficient to completely surround the inner vessel with an air gap of approximately $1\frac{1}{2}$ in. between the two vessels at all points. It supports the inner vessel and convection shield in its exact center by means of six $\frac{1}{4}$-in. pointed bakelite rods.

4. *Cover for Outer Vessel.*—Brass, chromium-plated and polished on both sides. It is fitted with four chimneys, for the following parts: (*a*) the lead wires of the resistance thermometer and heater coil, (*b*) an introductory tube for the cement sample, (*c*) the shaft of the propeller stirrer, and (*d*) a removable rod which connects the inner and outer covers when the calorimeter is disassembled. The chimneys are sufficiently long to permit the upper surface of the calorimeter to be immersed in the water bath to a depth of 1 in., with only the chimneys extending above the water. All permanent connections between the inner and outer covers are designed for low heat transfer.

5. *Convection Shield.*—Polished silver 0.003 in. thick, bisects the space between the inner and outer vessels on the sides and bottom. Its purpose is to minimize the transfer of heat by air convection currents.

6. *Stirrer.*—A multibladed platinum propeller driven by a constant-speed electric motor through a belt and pulleys. The propeller is located in the center of the annular heater coil which serves to direct the stirring downward. The propeller rotates at a high speed, 850 r.p.m., because the stirring must not only provide sufficient circulation to insure a uniform liquid temperature, but must also keep the sample agitated so that it may be rapidly attacked by the acid in the calorimeter.

7. *Heater Coil.*—Consists of manganin wire, with a resistance of approximately 75 ohms, wound in an annular case. The case is protected from acid attack by coatings of bakelite varnish and beeswax. In connection with a bank of storage cells, a precision potentiometer, and standard resistances, the heater coil permits the release and rapid dissipation of an accurately measured quantity of electrical energy in the calorimeter liquid.

8. *Resistance Thermometer.*—Four-coil bridge type developed by the U. S. Bureau of Mines.[1] This thermometer is well adapted to calorimetric measurements, as it is possible with it to make highly sensitive determinations of temperature change, in a small range, without the necessity of extreme sensitivity in the measuring instruments. With the potentiometer and mirror-type galvanometer used in this equipment it is easily possible to detect temperature changes of the order of 0.0001 C.

9. *Stop Watch.*—Having an error of less than two seconds in an hour. This watch is used principally in a measurement of the time interval during which electrical energy is being released by the heater

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[1] C. G. Maier, "Resistance Thermometers for Chemists," *Journal of Physical Chemistry,* Vol. XXXIV, December, 1930, p. 2860.

coil in the determination of the heat capacity of the calorimeter. For this purpose an automatic device has been developed which instantaneously starts or stops the watch when the switch which controls the heater current is manipulated. For the ordinary timing of calorimetric readings, a synchronous clock has been rebuilt to provide an audible signal (and warning signal) at one-minute intervals.

10. *Water Bath.*—An insulated galvanized-iron tank with a capacity of 35 gal. The bath is maintained at a constant temperature of 25 C. by means of a mercury regulator which operates through a vacuum-tube relay. Under the control of this device the bath temperature varies only a few thousandths of a degree during a day. The bath is stirred by a propeller incased in a tube and driven by an electric motor. Bath temperatures are measured with a calibrated Beckmann thermometer.

*Test Procedure:*

In preparation for a heat-of-solution test of either an unreacted or a partially hydrated cement sample, 1200.0 g. of 2.000-normal nitric acid and 10.0 ml. of chemically pure hydrofluoric acid are measured into the inner vessel, the whole being at a temperature close to 23 C. The calorimeter is then assembled and placed in the water bath, electrical connections to the heater coil and thermometer are soldered, and the calorimeter stirrer is started. In the meantime, the cement sample has been prepared in the form of a dry powder, as explained later, and is weighed into an introductory tube of special design, shown in Fig. 2. The introductory tube is placed in one of the chimneys of the outer vessel in order that the sample may reach the temperature of the bath prior to its introduction into the acid. When in place, the lower end of the introductory tube passes through a rubber gasket in the cover of the inner vessel and ends just clear of the acid directly above the stirrer propeller. A removable ground-glass needle valve permits the rate of introduction of the sample to be easily regulated.

After the assembly is complete, sufficient electrical energy is released in the heater coil to raise the acid temperature to 23.4 C., or 1.6 C. below the bath temperature. When the calorimeter has reached a constant rate of temperature change, readings are taken during a preliminary rating period. This and the final rating period each involve 10 to 20 temperature measurements made at one-minute intervals in order to determine the rate of change of calorimeter temperature caused by factors other than heat of solution. The introduction of the cement sample is then begun, and is continued at such a rate that the entire introduction consumes about five minutes.

During the introduction of the sample, the operator records simultaneous readings of time and calorimeter temperature at sufficiently frequent intervals to permit a time-temperature curve to be plotted. Similar readings are then continued at intervals of a few minutes until the temperature has reached a peak. As the total temperature rise in the calorimeter during the test is about 3 C., the peak temperature of the acid mixture is about 1.5 C. higher than that of the water bath, and the loss of heat resulting from this thermal head causes the calorimeter to cool gradually. When the solution of the sample is complete, as indicated by a substantially constant rate of cooling, readings are taken during the final rating period. The average test requires 30 to 60 minutes after the introduction of the sample.

*Calculations:*

The data obtained from the test are converted into terms of the heat of solution of the sample in calories per gram (ignited basis) of cement, in the following steps:

1. The rise in temperature of the acid mixture from the time at which introduction of the sample was begun to an arbitrarily chosen time a few minutes after the solution is completed is designated as the *uncorrected temperature rise* which takes place within the corresponding *elapsed time*.

2. The product of elasped time and *mean thermal head* between the acid mixture and water bath is determined by area measurements on a time-temperature diagram.

3. Two types of factors, other than heat of solution, affect the temperature rise in the calorimeter during test. The factors of one group, termed "heat of stirring," are constant during the test; those of the other group, termed "heat transfer," are proportional to the thermal head between the acid mixture and the bath. A coefficient for each group is computed from the rates of temperature change during the preliminary and final rating periods.

4. The product of the heat-of-stirring coefficient, which is expressed in degrees Centigrade per minute, and the elapsed time in minutes provides a correction to the uncorrected temperature rise for the factors which are independent of thermal head.

5. The product of the heat-transfer coefficient, which is expressed as degrees Centigrade per minute per degree of thermal head, the elapsed time in minutes, and the mean thermal head in degrees Centigrade provides a correction to the uncorrected temperature rise for the factors which are proportional to the thermal head.

6. The *corrected temperature rise*, which results solely from the heat of solution of the sample, is obtained by applying these two corrections to the uncorrected temperature rise.

7. The product of the corrected temperature rise in degrees Centigrade and the *heat capacity* of the calorimeter and the acid mixture expressed in calories per degree Centigrade represents the heat of solution of the sample in calories. Rather than by calculation, this heat capacity is determined experimentally by releasing a measured quantity of electrical energy in the calorimeter by means of the heater coil and by measuring and correcting the resulting temperature rise. To the heat capacity thus obtained is added the calculated heat capacity of the sample.

This experimental determination of heat capacity has an important advantage over the most precise calculated determination. As the shape of the time-temperature curve and the amount of the corrected temperature rise are caused to be nearly the same in the experimental determination as in a heat-of-solution test, several indeterminable errors such as those caused by thermometer lag, evaporation, circulation lag and the like, appear in the form of a "false" heat capacity, and if this false value is used in the calculation of the heat of solution these errors disappear.

8. A correction is applied to the heat of solution for the difference between the temperatures of sample and acid at the moment of introduction.

9. The heat of solution of the sample is divided by the ignited weight of the cement which it contains, in order to obtain the heat of solution of the sample expressed in calories per gram. The ignited weight of the calorimeter sample is determined by the ignition of an identical sample at 900 C. to a constant weight.

10. A partially hydrated sample requires one to two grams (ignited weight) of cement more than a corresponding unreacted sample, in order to obtain approximately the same temperature rise. This additional cement results in an increased concentration of sample in the solution, the effect of which has been experimentally determined to be a reduction in the heat of solution of approximately one calorie per gram. This amount must be added to the heat of solution of a partially hydrated sample, in order to make it comparable with that of the corresponding unreacted sample.

A partially hydrated sample also contains one to four grams of water. The heat of dilution of the acid mixture due to the addition of this water is negligible, as is also the effect of the reduction in strength of the acid.

11. The corrected heat of solution of a partially hydrated cement sample at any age is subtracted from the heat of solution of the corresponding unreacted cement, in order to determine the amount of heat generated by the cement from the time it was mixed with water until the time of test of the partially hydrated sample. This, the reported value, is termed the *heat of hydration.*

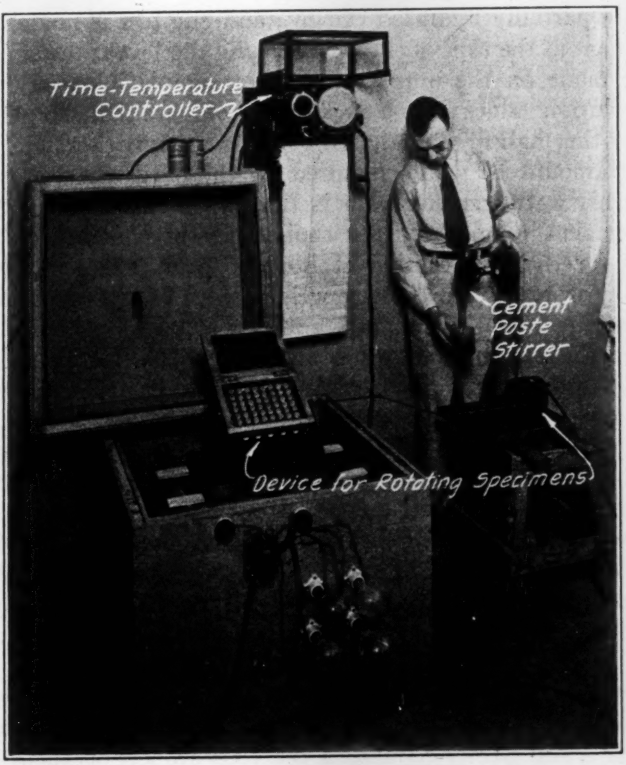

FIG. 4.—Variable-Temperature Chest and Time-Temperature Controller.

*Preparation and Testing of Samples:*

Cement samples are prepared for test in the following manner: Approximately 600 g. of cement, in its initial (unreacted) state, constitute one sample. This sample is carefully blended by rolling, and one-half is placed in a container which is sealed and stored at 70 F. (21 C.). This half is used in the unreacted cement test.

When a test on unreacted cement is to be made, about 20 g. of cement are removed from the container and the large particles are ground until the entire sample will pass a No. 100 sieve. The cement is then rolled and placed in a weighing bottle, and a 6.0000-g. calorimeter sample is weighed into an introductory tube and is ready to be tested. A loss-on-ignition sample of 1.0000 g. from the same weighing bottle is then weighed into a platinum crucible and is ignited to constant weight in a muffle furnace at 900 C. Usually two heat-of-solution tests are made on the unreacted cement at about the time of the 7-day test of the partially hydrated cement, and one test is made on unreacted cement at the time of the 6-month and the 1-year tests. Further tests are made on the unreacted cement sample if at any time its heat-of-solution values show a discrepancy of more than 0.3 per cent.

The other half of the original 600-g. sample is thoroughly mixed with the amount of water required by the particular water-cement ratio. This water-cement ratio is that which has been determined for the cement to give a fixed consistency in concrete of the mix proposed for the construction of Boulder Dam. The water-cement ratios vary from 0.51 to 0.63 by weight, with an average of 0.58 for the cements tested in this investigation.

After the cement and water have been thoroughly mixed, ten or more 4-dram glass vials are filled with the paste. The vials are stoppered with corks, which are held in place with screw caps. They are then placed in the rotator shown in Fig. 4, the rotator is enclosed in a variable-temperature chest (to be described later) and the vials are rotated end over end at a speed of 2 r.p.m. for 24 hr. This procedure prevents settlement of the cement from the relatively large quantity of water used in the paste. After 24 hr. the vials are removed from the rotator and placed in 1-pint Mason jars containing a few ounces of water, and the jars are returned to the curing chest.

For a large number of auxiliary tests, a water-cement ratio of 0.40 by weight has been used, in order to eliminate the necessity for rotation of the vials. With this relatively low water-cement ratio, settlement is sufficiently reduced if the vials are left on their sides for the first 24 hr. of curing. The heat-of-solution value obtained with this amount of water is greater than with the normal water-cement ratio, but satisfactory conversion factors have been determined which make it possible to calculate the correct heat-of-solution value for the water-cement ratio used in concrete. To simplify the curing of specimens, this water-cement ratio of 0.40 has been specified for cement acceptance tests for Pine Canyon Dam and Boulder Dam.

To prepare the heat-of-solution sample of partially hydrated cement, a vial of cement paste is removed from the curing chest, the glass is broken away from the cylinder of hardened paste, and the cylinder is rapidly pulverized with a heavy porcelain mortar and pestle until it all passes a No. 100 sieve. The partially hydrated cement is then rolled and placed in a weighing bottle.

The size of a calorimeter sample of partially hydrated cement is estimated in each case, and is governed by the desire to produce a temperature rise in the calorimeter of approximately 3 C., which is about the rise obtained with 6.0000 g. of an unreacted cement. The sample, which varies in amount from 8 to 11 g., is then weighed with equal precision by difference from the weighing bottle into an introductory tube, and is ready to be tested in the calorimeter. The loss-on-ignition sample, which is about 2.5 g. in this case, is weighed into a platinum crucible in the same manner. The difference method of weighing is essential, as the partially hydrated cement contains a large quantity of free water and changes weight rapidly when it is exposed to the air. In order that no further reaction may take place after the partially hydrated sample is removed from the comparatively high temperature of the curing chest, the time between removal and test is kept at a minimum and is never allowed to exceed one hour.

A heat-of-solution test is made on each of two separate samples of neat-cement paste at the ages of 7 days, 28 days, 3 months, 6 months, and 1 year. Additional tests at any age are made if the two tests show a discrepancy of more than 0.5 per cent. Inasmuch as these tests of partially hydrated cement are made on separate vials of the neat cement, any difference in the water content or curing of the specimens causes an error, and the correlation cannot be expected to be as close as in the case of tests on unreacted cement.

## CURING OF SPECIMENS

### *High-Insulation Calorimeters:*

It has been determined that the heat generation of cement varies widely with the temperature of curing, particularly at the early ages of hydration. It follows that the temperature history of test specimens of neat cement must approximate that of corresponding concrete. For each cement under test, therefore, an approximate time-temperature relation for mass concrete has been experimentally determined to serve as a guide in the curing of heat-of-solution (and other) specimens.[1] These data have been obtained in a set of six "high-insulation" calorimeters, using concrete specimens.

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[1] It has been shown, however, that a simplified curing condition, one day at 70 F. (21 C.) and thereafter at 100 F. (38 C.), provides satisfactory heat-generation values at the ages of 7 and 28 days for low-heat cements. This curing condition has been specified for cement acceptance tests for Pine Canyon Dam and Boulder Dam.

The high-insulation calorimeter consists of a 20 by 20-in. cylindrical compartment surrounded by a minimum thickness of 1 ft. of kapok. In such a calorimeter the heat transfer from specimen to surroundings, and *vice versa*, is sufficiently slow to represent a small percentage of the total heat generated by the specimen. Its only requisites of high insulation, low heat capacity, and a constant temperature of the surrounding air are capable of being met without high construction cost or expensive control equipment. The six calorimeters of this investigation are housed in a steam-heated room thermostatically controlled at a temperature of 90 F. (32 C.).

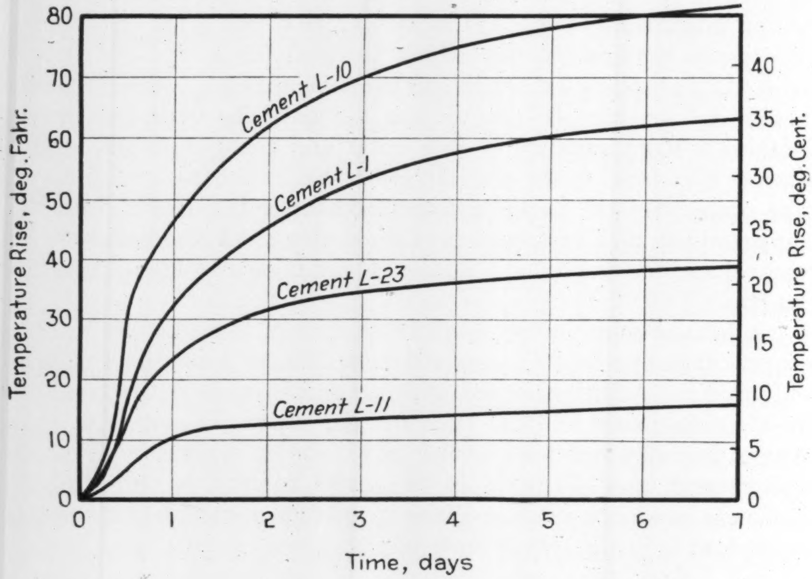

FIG. 5.—Typical Time-Temperature Relationships of Mass Concrete Containing Different Cements.

Initial temperature approximately 70 F. (21 C.).

A high-insulation-calorimeter specimen consists of approximately 400 lb. of concrete which is cast at 70 F. (21 C.) in an 18 by 18-in. cylindrical metal container. Two resistance thermometers are embedded in the concrete. The cement content of the concrete mix used in these specimens is such that the ratio of the quantity of cement to the heat capacity of the specimen and inner portions of the calorimeter is equal to the ratio which will exist for the proposed mix and materials for Boulder Dam.

As soon as the specimen is cast, the container is sealed and placed in the compartment of a high-insulation calorimeter. Simultaneously with the placing of test specimens (usually five) in the calorimeters, another similar cylinder of aged concrete, inert as to heat generation and at a temperature different from that of the room, is placed in the sixth calorimeter to serve as a calibration specimen which will indicate the amount and rate of heat transfer between room and specimens.

Temperature observations to the nearest 0.1 F. (0.06 C.) are made on the specimens at frequent intervals for a period of seven days, by means of the resistance thermometers. Using the information gained from the temperature change of the inert calibration specimen, the observed temperature rises of the specimens under test are corrected for heat transfer. In this manner, approximate relationships between time and temperature are obtained for the first seven days of hydration. The rate of heat generation of cement after the first seven days is comparatively slow, and no appreciable error is incurred if the temperature history of heat-of-solution specimens follows an estimated rate of change after the age of seven days.

Typical early time-temperature relationships are shown in Fig. 5 for cements of a wide range of heat generation. Cement L-1 is a normal portland cement, and L-23 is approximately of the composition and fineness specified as the low-heat cement for Boulder Dam. It is noted that for the conditions of this test the greatest rate of temperature rise of each cement occurs between 8 and 12 hours after casting.

*Variable-Temperature Chests:*

In order that the time-temperature curves obtained from the high-insulation calorimeters may be duplicated in the curing of specimens for heat-of-solution, strength, volume-change and durability tests, it is necessary to employ a curing compartment in which the temperature may be varied according to a definite program. For this purpose six variable-temperature chests, one of which is shown in Fig. 4, are used. These chests are roughly cubical in shape, with the length of a side being about 3 ft. The walls, bottom, and removable top are made up of a 1-in. thickness of kapok held in place by two sheets of $\frac{1}{2}$-in. celotex on a wooden frame. The heating elements are eight 50-watt lamps, spaced evenly around the bottom of the box near the sides. These lamps are operated by a relay, and their output is regulated by series lamps of suitable resistance placed on the outside of the chest. A baffle placed between the lamps and the storage space at the center of the chest promotes an efficient natural circulation of air.

When the temperature of one of these chests is being held at a constant value or is being changed very slowly, the temperature is controlled by a thermostat of the type used in the manually controlled adiabatic calorimeters, previously described. A thermostat of this type will maintain the temperature at which it is set, but it must be manually adjusted when the control point is changed. It is obvious that in order to duplicate a time-temperature program of the type shown in Fig. 5 with the desired limit of error of 1 F. (0.6 C.), almost continuous manipulation of the thermostat would be required during the first day or so of curing.

*Time-Temperature Controllers:*

In order to control the temperature of the chests during the important early stage of curing, two recording time-temperature controllers have been especially modified for this installation. The feature of these controllers is that the control point is automatically changed to correspond to the time-temperature history of any cement.

One of the instruments is shown in Fig. 4. On the front are two disks, one of which regulates the temperature-control point. Ordinarily an operator sets this disk at the desired control temperature, and if at any time the temperature in the chest differs from this set temperature, the relay is actuated as it is under thermostatic control. Electrically the instrument is a Wheatstone bridge, and measures temperature by means of a copper resistance thermometer which can be seen in the center of the cover of the chest in Fig. 4. It is sensitive to a temperature change of less than 0.5 F. (0.3 C.).

The other disk on the front of the controller is driven by the controller motor at a rate of one revolution in 12 hr. The periphery of this disk is calibrated in hours and minutes, and at intervals of 5 minutes there are threaded holes. When a steel peg screwed into one of these holes passes the common centerline of the two disks it strikes a star gear which is connected through a gear train to the temperature-control disk, and the control point is advanced precisely 1 F. (0.6 C.).

To prepare the time disk to control the curing temperature for specimens of a given cement, the time-temperature curve which has been determined for that cement in a high-insulation calorimeter is plotted to a large scale, and the time of each degree Fahrenheit change is noted to the nearest five minutes. A peg is screwed into the time disk at the time of each of these changes, and the disk is then placed on the controller.

When the specimens have been prepared and placed in the curing chest, the controller motor is started and the temperature of the chest is automatically carried from the initial temperature of 70 F. (21 C.) through the desired program without further attention for 12 hr. At this time, a disk pegged for the next 12 hr. is substituted for the first time disk. After 24 hr. have elapsed, the gear ratio of the time disk is changed to one revolution per day, and thereafter a disk calibrated for 24 hr. is used. In this manner the proper temperature program is followed for the first week with a single change of disks each day. After a week the temperature changes very slowly, and the chest is put under thermostatic control.

### *Constant-Temperature Chests:*

Three constant-temperature chests of large capacity are maintained at temperatures a few degrees apart, near the extreme values for the different cements, for storage of specimens at later ages. The constant-temperature chests are heated by a hot-water circulating system. The temperatures are controlled by thermostats which operate magnetic valves to regulate the flow of hot water.

## USE OF CALORIMETERS AND CURING CHAMBERS

The functions of the various calorimeters and curing chambers in the general program of investigation are interdependent. For the preliminary curing of neat-cement specimens for the heat-of-solution tests, the approximate time-temperature history of each cement (in mass concrete) is determined by means of a concrete specimen in a *high-insulation calorimeter*, and the specimens are cured according to this history in a *variable-temperature chest.*

*Heat-of-solution* tests are made at intervals on the partially hydrated neat-cement specimens, in order to determine the heat of hydration up to the age of test.

For selected cements only, control and check tests are made by the temperature-rise method, employing concrete specimens in *adiabatic calorimeters* which provide a continuous record of temperature rise in mass concrete and which are therefore basic for the purposes of this investigation. Also, cements which contain appreciable amounts of ingredients or admixtures insoluble in the dilute nitric- and hydrofluoric-acid mixture of the heat-of-solution calorimeter are necessarily tested in the adiabatic calorimeters.

### *Relation Between Temperature Rise and Heat Generation:*

It should be emphasized that the adiabatic calorimeters are designed to measure the *temperature rise* in mass-concrete specimens, while the heat-of-solution calorimeter is designed to measure the *amount of heat* generated by a unit quantity of cement. Although in structural design the temperature rise is the primary information desired, the amount of heat must be known if it is desired to calculate in advance the probable distribution of temperature within the mass, the rate of cooling, and (specifically in connection with Boulder Dam) the amount of refrigeration required to reduce the temperature of the concrete to normal at a given age. Conversion into heat units is also necessary if the data are to be used in connection with other aggregates, mixtures, or conditions of concrete manufacture. Such conversion requires a knowledge of the specific heat of the concrete, a quantity which varies with temperature.

Values of heat generation computed directly from observed temperature rises in an adiabatic calorimeter are less than corresponding values determined by the heat-of-solution method. The measurement of temperature rise in the adiabatic calorimeter begins approximately one half hour after the cement is wetted, corresponding roughly to construction conditions, and any heat generated prior to this time is not included in the measurement.

When due allowance is made for the differences in test conditions, the results of check tests by the two methods are in satisfactory agreement.

## RESULTS OF TESTS

The test data obtained in this investigation are providing the basis for study of the effect of chemical composition, fineness of grinding, and method of manufacture of cement upon the heat of hydration of cement in concrete. The major results obtained during the first $1\frac{1}{2}$ years of the investigation, with tentative conclusions based thereon, have been recently reported.[1] In general, it is shown that the heat generation of cement under given conditions of curing is greatly affected by the chemical composition of the cement and is affected to a lesser degree by the fineness of grinding and by the water-cement ratio of the mix. Without exceeding limits of portland-cement compositions practicable of commercial manufacture, the range in total heat generation is perhaps 30 per cent above and below a normal value, and the rate at which heat is generated may be varied within rather wide limits.

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[1] R. E. Davis, R. W. Carlson, G. E. Troxell and J. W. Kelly, "Cement Investigations for Hoover Dam," paper presented at Twenty-ninth Annual Convention, Am. Concrete Inst. (1933).

# DISCUSSION

MESSRS. R. W. STENZEL[1] AND R. G. FOLSOM[2] (*presented in written form*).—The calorimeter in use by the City of Pasadena Water Department for testing the cement being furnished for the Pine Canyon Dam is patterned after that described by Biddle and Kelly and has been found quite satisfactory. A few modifications in design and procedure have been made in the interest of improved operation. The base of the inner vessel was made spherical to provide more effective circulation and thus allow a reduction in the stirring speeds (to 650 r.p.m.). This modification also permits the sample to be introduced rapidly (over a period of 15 seconds instead of 5 minutes), thereby decreasing the time of operation and providing uniform time-temperature curves. A double pulley allows the stirring rate to be increased during and shortly after the introduction to avoid formation of lumps which dissolve extremely slowly.

Some of the low-heat cements have been very resistant to complete solution by the acid mixture described, so recourse was had to doubling the amount of the hydrofluoric acid in the charge (20 ml. instead of 10 ml.). This results in a marked reduction of the reaction time as well as insuring the complete solution of cement, which is essential.

The Pasadena calorimeter is operated at a bath temperature of 30 C. which results in a different heat of solution for the cement, but gives, of course, the same heat-of-hydration results as operation at any other suitable temperature. For the low-heat cements investigated, the temperature coefficient of the heat of solution is $-0.3$ cal. per gram per degree Centigrade.

The usual heat capacity determination by means of a measured electrical energy input is somewhat tedious so that it was thought desirable to use a chemical reaction as a secondary standard. Ignited zinc oxide was found to be a suitable material, being only slightly hygroscopic, and having a heat of solution per gram about half that of cement. The chemically pure products of different manufacturers gave the same heat of solution.

It has been our experience that the limiting factor in the accuracy of the method is associated with the operations of hydrating the cement and preparing the hydrated sample for the calorimeter. As this technique is improved, the increased precision should lead to valuable correlation of heats of hydration and other physical and chemical properties.

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# FURTHER STUDIES OF PAVING CONCRETE

## INVESTIGATION OF THE EFFECT OF VIBRATION AND DELAYED FINISHING ON THE QUALITY OF PAVEMENT SLABS

By F. H. Jackson[1] and W. F. Kellermann[2]

## Synopsis

This paper presents the results of a series of tests conducted recently by the U. S. Bureau of Public Roads on a number of full-size concrete pavement sections, placed and finished under actual working conditions. The general procedure was similar to that outlined in connection with the report of an earlier series presented before the Society in 1931.[3]

In the earlier tests it was determined quite definitely that pavement concrete having a slump less than 2 in. could not be placed satisfactorily by the methods of finishing now in common use. The present paper reports the results of tests to determine the effect of vibrating the concrete during the finishing operation as well as a method of delayed finishing for the purpose of removing excess water known as the "Johnson method."

In general the tests indicate that by using vibratory equipment of the type employed it should be possible satisfactorily to place and finish drier concrete as well as concrete containing considerably more coarse aggregate than by the methods now in common use.

The indications also are that the use of the so-called "Johnson method" of finishing will increase the density and strength of the concrete pavement slabs by eliminating a larger quantity of excess water before final consolidation than is accomplished by the methods now in common use.

In the Society's *Proceedings*, Vol. 31, Part II, p. 457 (1931), there was published a report describing the results of an investigation conducted by the U. S. Bureau of Public Roads to determine the effect of certain controllable variables on the quality of pavement concrete. The test procedure used in this work differed from the conventional laboratory investigation in that observations and tests were made on actual pavement sections, placed and finished under working conditions rather than on small-size test specimens. In this way it was possible to measure the effect of variations in "workability" by studying the quality and uniformity of the concrete in the finished structure rather than by attempting to investigate this indefinite property through the use of some arbitrary laboratory test such as flow, penetration, etc.

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[3] F. H. Jackson, "Studies of Paving Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 31 Part II, p. 457 (1931).

In the tests which have already been reported, a study was made of the effect of varying the quantity of coarse aggregate in the mix for various types and gradings of coarse aggregate and for various consistencies. The concrete was mixed and placed in accordance with accepted construction practice, which included finishing by the use of the conventional type power-driven screeds, with and without tamper attachment. These initial tests developed certain information of value. For instance, it was quite definitely determined that with the methods of machine finishing now commonly employed a consistency corresponding to a slump of at least 2 in. must be used if honeycomb is to be avoided. Of course, such a concrete does not have as high a potential strength and density as a somewhat drier mix. Actually, however, it was found that both strength and uniformity were improved by the use of the wetter consistency. This is due to the fact that the segregation and honeycombing which accompanies the use of the drier mix actually tends to lower the strength of the pavement slab to a point considerably below the strength which would be expected theoretically from the water-cement ratio - strength law. This means that under present construction practice concretes mixed in the proportions used in paving work and with normal aggregates are not "workable" as paving mixes when slumps less than 2 in. are used. In other words, it is not possible to take advantage of the higher strength and density which should accompany the use of a lower water content than is required to give a 2-in. slump.

A realization of this fact led naturally to a consideration of methods of placing which would permit the use of drier mixes without sacrificing strength or uniformity of strength. The use of vibratory screeds for this purpose presented interesting possibilities and it was decided to continue the experiments using a standard finishing machine equipped with vibrators mounted directly on the screeds. The present paper discusses the results of these tests, as well as another series in which a method of removing excess water from the concrete after it was deposited on the subgrade was investigated. In both cases the ultimate object was the same—the production of a pavement slab of uniform quality with a lower net water content and consequently higher strength and greater density than would be possible with a standard finishing machine.

For the purpose of discussion, the two series of tests are treated separately. The results obtained by the use of vibrators comprise series A, and the discussion of the tests which involved the principle of delayed finishing for the purpose of removing excess water, otherwise known as the "Johnson method," series B. In both cases the general testing procedure outlined in the 1931 report was followed, except for the finishing operations, and the fact that the slabs were cast on the bare subgrade whereas, in the former tests, the subgrade was covered with tar paper before placing the concrete.

The same general types of crushed limestone, gravel and blast-furnace slag were used as coarse aggregate. However, in the tests reported herein the aggregates were used in two separated sizes only instead of three as in the earlier investigation. Two sands, differing considerably in gradation, were also used instead of one as in the first series. In this way it was possible, in the case of series A, to investigate six combinations of aggregates varying as to character and gradation; that is, crushed stone, gravel and slag each in combination with a fine sand and a coarse sand. Blast-furnace slag was omitted from the Johnson series and in its place was substituted a sand-gravel combination conforming in grading to the so-called "Platte River Gravel" used in Nebraska, Kansas and Iowa. The essential test data as regards the aggregates used in this investigation are given in Table I. The same cement, a standard portland cement

TABLE I.—PROPERTIES OF AGGREGATES.

| | Coarse Sand | Fine Sand | Gravel | Crushed Stone | Crushed Slag | Platte River Gravel |
|---|---|---|---|---|---|---|
| SIEVE ANALYSIS | | | | | | |
| Retained on $1\frac{1}{2}$-in. sieve, per cent | .... | .... | 21 | 17 | 4 | .... |
| Retained on $\frac{3}{4}$-in. sieve, per cent | .... | .... | 68 | 76 | 65 | .... |
| Retained on $\frac{3}{8}$-in. sieve, per cent | .... | .... | 90 | 95 | 99 | 11 |
| Retained on No. 4 sieve, per cent | 5 | 0 | 99 | 99 | 100 | 31 |
| Retained on No. 8 sieve, per cent | 24 | 2 | 100 | 100 | 100 | 46 |
| Retained on No. 14 sieve, per cent | 36 | 8 | 100 | 100 | 100 | 55 |
| Retained on No. 28 sieve, per cent | 54 | 30 | 100 | 100 | 100 | 67 |
| Retained on No. 48 sieve, per cent | 83 | 77 | 100 | 100 | 100 | 89 |
| Retained on No. 100 sieve, per cent | 96 | 96 | 100 | 100 | 100 | 98 |
| Fineness modulus | 2.98 | 2.13 | 7.78 | 7.87 | 7.68 | 3.97 |
| PHYSICAL PROPERTIES | | | | | | |
| Apparent specific gravity | 2.66 | 2.65 | 2.54 | 2.78 | 2.32 | 2.63 |
| Absorption, per cent | 0.82 | 0.70 | 1.20 | 0.36 | 4.25 | 0.91 |
| Decantation loss, per cent | 2.7 | 2.1 | .... | .... | .... | .... |
| Voids, per cent[a] | 37 | 41 | 32 | 40 | 43 | 31 |
| Wear, per cent[b] | .... | .... | 9.6 | 3.4 | 7.7 | .... |

[a] Dry-rodded.

[b] Abrasion tests conducted in accordance with methods applicable in each case.

meeting all requirements of the specifications of this Society, was used throughout. The proportions, consistency, etc., of the concrete for series A are shown in Table II with similar data for series B in Table III.

## METHODS OF TESTING

### *Strength Tests:*

As in the earlier tests, the flexural strength of each 9-ft. pavement section was obtained by averaging the results of flexure tests on four slabs removed from the section and tested in accordance with the method described in the first report. The actual test slabs were 27 in. in width by 60 in. in length by 7 in. in depth. They were loaded at the third points of a 54-in. span. The same general procedure as regards control specimens was followed also in the present series. These specimens consisted of beams tested at the age of 7 months and cylinders tested at the age of 9 months. These periods corresponded to the ages at which the pavement slabs and cores drilled therefrom were tested. All strength tests were made on saturated specimens.

### *Absorption and Density Tests:*

Four 6-in. cores were drilled from each test section, two each from two of the half slabs remaining after the flexure tests. In drilling the cores, considerable care was taken to select portions of the slabs free from honeycomb, in order to insure the use of sound, homogeneous cores for testing purposes. Two of the cores, one from each slab, were tested in compression while the other two were tested for absorption and density. The following procedure was employed in making the absorption and density tests: The cores were first dried to constant weight in an electric oven where the temperature was maintained at approximately 150 C. They were then cooled, weighed, immersed in water and boiled for 5 hr. The specimens were then allowed to cool for 18 hr., after which they were removed from the water, surface dried and weighed. They were then weighed in water and from the three weights thus obtained the absorption, weight per cubic foot and specific gravity were computed.

Values for the density of the hardened concrete were obtained by dividing the weight of a given volume of the concrete by the theoretical weight of solids in the same volume as calculated from the proportions and specific gravities of each of the constituent materials. In the case of series B, a value which may be termed the theoretical density of the plastic concrete was also obtained, as the ratio of the volume of solids (cement and aggregate) to the total volume of concrete calculated on the assumption that the freshly mixed concrete is free from air voids. This, of course, is not absolutely true. However, tests of concrete similar to that used in series B, where the difference in consistency of the concretes finished by the two methods was not great (ranging from about 1½ in. to 3 in. in slump), have shown that the error introduced by not correcting for air voids is small and reasonably constant and therefore may be neglected. On this basis a comparison of the two values for density may be used as a rough measure of the amount of water loss taking place during consolidation. In other words, it may be assumed that the increase in density of the hardened concrete as compared with the density of the plastic concrete is due to the elimination of water only. The significance of these values will be discussed in that portion of the report dealing with series B.

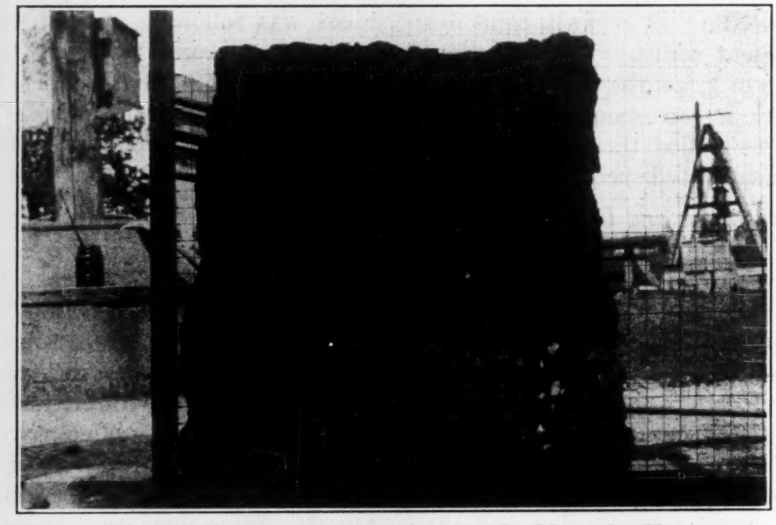

FIG. 1.—Use of Grid with 1-in. Square Openings for Measuring Honeycomb.

*Determination of Honeycomb:*

The extent and distribution of visible honeycomb in the test slabs was carefully determined by measurement of the fractured slabs remaining after the flexure test. The actual area of honeycomb on the bottom of each 27 by 60-in. slab was determined by means of a wire grid having 1-in. square openings laid upon the surface (see Fig. 1). The amount of honeycomb in the cross-section at the break was determined by plotting the honeycomb area on cross-section paper. These procedures provided accurate methods of determining the percentage of the bottom surface which contained honeycomb areas exceeding approximately $\frac{1}{4}$ in. in depth, and the percentage of honeycomb at the break. The average percentage of honeycomb for an entire section as used in the discussion is the average of eight determinations, one on the bottom and one at the plane of fracture for each of the four test slabs composing the section.

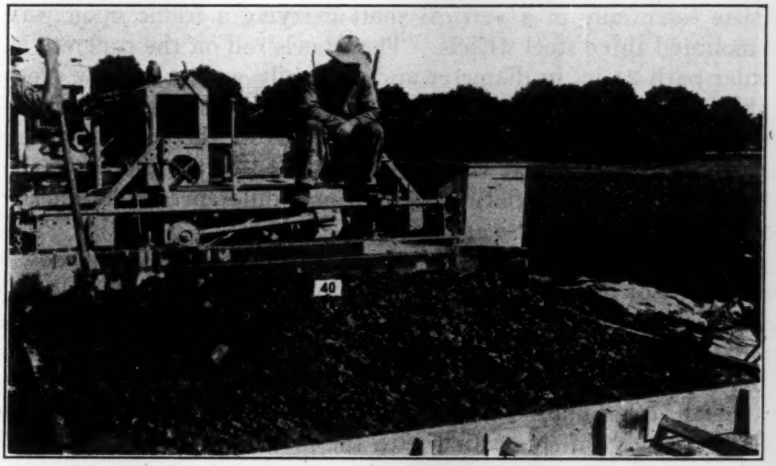

FIG. 2.—Vibrating Method, Before Finishing.

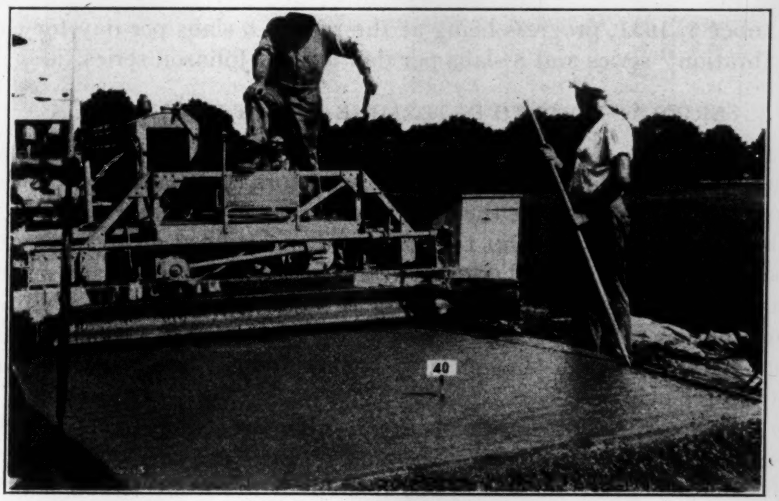

FIG. 3.—Vibrating Method, After Finishing.

*Determination of Surface Wear:*

Determinations of the surface hardness of the test slabs were made with a special device designed by the Bureau and described in detail in *Public Roads* for July, 1929. The hardness testing machine consists essentially of a vertical shaft carrying a frame upon which are mounted three steel wheels. The wheels roll on the concrete in a circular path 21 in. in diameter and gradually wear a narrow groove in the surface, the depth of which, after a given number of revolutions, is used as a measure of the hardness of the mortar surface. Care is taken to see that the wear is confined to the mortar surface only so as to avoid variations due to any difference in hardness of the mortar and the coarse aggregate.

The results of the strength tests, together with data showing the amount of honeycombing existing in each test slab, the density, absorption and resistance to wear of the concrete, form the basis of comparison used in this report to determine the effect of the methods of finishing on quality.

In all, one hundred and six 9-ft. sections were cast during this investigation, 66 in connection with the investigation of the effect of vibration and 40 in connection with the tests of the Johnson method. The first slabs were cast on September 10, 1931, and the last on October 5, 1931, progress being at the rate of 6 slabs per day for the "vibration" series and 8 slabs per day for the Johnson series.

## SERIES A—TESTS TO DETERMINE EFFECT OF VIBRATION

For series A a standard double screed finishing machine was equipped with three high-frequency vibrators, two mounted on the front screed and one on the rear screed (see Figs. 2 and 3). Each vibrator consisted of a $\frac{1}{3}$-hp. electric motor operating at an approximate speed of 3600 r.p.m. There was attached to the rotor of each motor an eccentric weight which, revolving at high speed, imparted a vibratory motion to the screed. The motors were operated with current supplied from a gasoline-electric generator unit mounted on the finishing machine.

The only modification of the finishing machine as ordinarily used, other than the installation of the vibrators, was the use of a "bull nose" strike-off which was riveted to the front screed. This device was furnished and recommended by the manufacturer of the finishing machine on the theory that the rounded surface of the "bull nose" would tend to consolidate as well as strike off the surface of the concrete.

In general, the finishing machine was operated at about the

TABLE II.—DATA ON MIXES, SERIES A.

| Section[a] | Proportions by Volume, Dry-Rodded | Cement Factor, sacks per cu. yd. | Water-Cement Ratio, Corrected | Ratio, $\frac{b}{b_o}$ | Slump, in. |
|---|---|---|---|---|---|
| COARSE SAND AND GRAVEL (ROUND 1) | | | | | |
| No. 1 S | 1:1.6:3.45 | 5.9 | 0.75 | 0.75 | 2¼ |
| No. 2 V | 1:1.6:3.45 | 5.9 | 0.69 | 0.76 | 1 |
| No. 3 V | 1:1.6:3.45 | 6.0 | 0.65 | 0.77 | 0 |
| No. 4 V | 1:1.6:3.70 | 5.7 | 0.74 | 0.78 | 2½ |
| No. 5 V | 1:1.6:3.95 | 5.5 | 0.74 | 0.80 | 1¼ |
| No. 6 V | 1:1.6:4.20 | 5.3 | 0.73 | 0.82 | 1 |
| FINE SAND AND GRAVEL (ROUND 1) | | | | | |
| No. 7 S | 1:1.5:3.45 | 6.1 | 0.73 | 0.77 | 1½ |
| No. 8 V | 1:1.5:3.45 | 6.1 | 0.68 | 0.78 | ½ |
| No. 9 V | 1:1.5:3.45 | 6.1 | 0.67 | 0.78 | ¼ |
| No. 10 V | 1:1.5:3.70 | 5.8 | 0.74 | 0.80 | 1¼ |
| No. 11 V | 1:1.5:3.95 | 5.6 | 0.73 | 0.82 | 1 |
| No. 12 V | 1:1.5:4.20 | 5.5 | 0.72 | 0.85 | 0 |
| COARSE SAND AND STONE (ROUND 1) | | | | | |
| No. 13 S | 1:1.9:3.3 | 6.1 | 0.75 | 0.75 | 1½ |
| No. 14 V | 1:1.9:3.3 | 6.2 | 0.69 | 0.76 | ¾ |
| No. 15 V | 1:1.9:3.3 | 6.2 | 0.67 | 0.76 | 0 |
| No. 16 V | 1:1.9:3.55 | 5.9 | 0.75 | 0.78 | 1½ |
| No. 17 V | 1:1.9:3.80 | 5.7 | 0.74 | 0.81 | 1½ |
| No. 18 V | 1:1.9:4.05 | 5.5 | 0.74 | 0.83 | 1¼ |
| FINE SAND AND STONE (ROUND 1) | | | | | |
| No. 19 S | 1:1.7:3.3 | 6.4 | 0.76 | 0.78 | 1½ |
| No. 20 V | 1:1.7:3.3 | 6.5 | 0.70 | 0.79 | 1 |
| No. 21 V | 1:1.7:3.3 | 6.5 | 0.68 | 0.79 | ¾ |
| No. 22 V | 1:1.7:3.55 | 6.2 | 0.75 | 0.81 | 1¼ |
| No. 23 V | 1:1.7:3.8 | 6.0 | 0.75 | 0.84 | 1¼ |
| No. 24 V | 1:1.7:4.05 | 5.8 | 0.75 | 0.87 | ¾ |
| COARSE SAND AND SLAG (ROUND 1) | | | | | |
| No. 25 S | 1:2.1:3.2 | 6.2 | 0.75 | 0.73 | 2 |
| No. 26 V | 1:2.1:3.2 | 6.3 | 0.67 | 0.74 | 1 |
| No. 27 V | 1:2.1:3.2 | 6.3 | 0.63 | 0.75 | ½ |
| No. 28 V | 1:2.1:3.45 | 6.0 | 0.73 | 0.76 | 1¼ |
| No. 29 V | 1:2.1:3.7 | 5.8 | 0.72 | 0.80 | 1 |
| No. 30 V | 1:2.1:3.95 | 5.7 | 0.70 | 0.83 | ¾ |
| FINE SAND AND SLAG (ROUND 1) | | | | | |
| No. 31 V | 1:1.9:3.2 | 6.4 | 0.79 | 0.76 | 1 |
| No. 32 V | 1:1.9:3.2 | 6.3 | 0.83 | 0.75 | 2 |
| No. 33 V | 1:1.9:3.2 | 6.4 | 0.75 | 0.76 | ½ |
| No. 34 V | 1:1.9:3.45 | 6.1 | 0.82 | 0.78 | 1 |
| No. 35 V | 1:1.9:3.7 | 6.0 | 0.81 | 0.82 | 1 |
| No. 36 V | 1:1.9:3.95 | 5.8 | 0.79 | 0.85 | ½ |
| COARSE SAND AND GRAVEL (ROUND 2) | | | | | |
| No. 37 S | 1:1.6:3.45 | 6.0 | 0.67 | 0.76 | 2¼ |
| No. 38 V | 1:1.6:3.45 | 6.0 | 0.61 | 0.77 | 1 |
| No. 39 V | 1:1.6:3.45 | 6.1 | 0.58 | 0.78 | ¼ |
| No. 40 V | 1:1.6:3.7 | 5.8 | 0.66 | 0.79 | 1¼ |
| No. 41 V | 1:1.6:3.95 | 5.6 | 0.66 | 0.81 | 1¼ |
| No. 42 V | 1:1.6:4.2 | 5.4 | 0.66 | 0.84 | 1 |

[a] S=standard finish. V=vibrated.

TABLE II.—(*Continued*).

| Section[a] | Proportions by Volume, Dry-Rodded | Cement Factor, sacks per cu. yd. | Water-Cement Ratio, Corrected | Ratio, $\frac{b}{b_o}$ | Slump, in. |
|---|---|---|---|---|---|
| FINE SAND AND GRAVEL (ROUND 2) | | | | | |
| No. 43 V | 1:1.5:3.45 | 6.1 | 0.67 | 0.78 | $1\frac{1}{4}$ |
| No. 44 S | 1:1.5:3.45 | 6.1 | 0.72 | 0.78 | $2\frac{1}{2}$ |
| No. 45 V | 1:1.5:3.45 | 6.2 | 0.61 | 0.80 | 1 |
| No. 46 V | 1:1.5:3.7 | 5.9 | 0.71 | 0.80 | 2 |
| No. 47 V | 1:1.5:3.95 | 5.7 | 0.71 | 0.83 | $1\frac{1}{2}$ |
| No. 48 V | 1:1.5:4.2 | 5.5 | 0.70 | 0.85 | $1\frac{1}{4}$ |
| COARSE SAND AND STONE (ROUND 2) | | | | | |
| No. 49 S | 1:1.9:3.3 | 6.1 | 0.75 | 0.75 | $1\frac{3}{4}$ |
| No. 50 V | 1:1.9:3.3 | 6.2 | 0.69 | 0.76 | $1\frac{1}{4}$ |
| No. 51 V | 1:1.9:3.3 | 6.2 | 0.65 | 0.76 | $\frac{1}{4}$ |
| No. 52 V | 1:1.9:3.55 | 5.9 | 0.75 | 0.77 | $1\frac{3}{4}$ |
| No. 53 V | 1:1.9:3.8 | 5.7 | 0.75 | 0.80 | $1\frac{3}{4}$ |
| No. 54 V | 1:1.9:4.05 | 5.5 | 0.75 | 0.83 | 1 |
| FINE SAND AND STONE (ROUND 2) | | | | | |
| No. 55 S | 1:1.7:3.3 | 6.4 | 0.76 | 0.78 | 2 |
| No. 56 V | 1:1.7:3.3 | 6.5 | 0.70 | 0.79 | $1\frac{1}{2}$ |
| No. 57 V | 1:1.7:3.3 | 6.5 | 0.66 | 0.80 | $\frac{1}{2}$ |
| No. 58 V | 1:1.7:3.55 | 6.2 | 0.76 | 0.81 | 2 |
| No. 59 V | 1:1.7:3.8 | 6.0 | 0.76 | 0.84 | $1\frac{1}{2}$ |
| No. 60 V | 1:1.7:4.05 | 5.8 | 0.76 | 0.86 | $\frac{1}{4}$ |
| COARSE SAND AND SLAG (ROUND 2) | | | | | |
| No. 61 S | 1:2.1:3.2 | 6.2 | 0.75 | 0.73 | $2\frac{3}{4}$ |
| No. 62 V | 1:2.1:3.2 | 6.3 | 0.66 | 0.74 | $1\frac{1}{2}$ |
| No. 63 V | 1:2.1:3.2 | 6.3 | 0.62 | 0.75 | $\frac{1}{2}$ |
| No. 64 V | 1:2.1:3.45 | 6.0 | 0.73 | 0.75 | $1\frac{1}{2}$ |
| No. 65 V | 1:2.1:3.7 | 5.8 | 0.72 | 0.80 | $1\frac{1}{4}$ |
| No. 66 V | 1:2.1:3.95 | 5.7 | 0.70 | 0.83 | 1 |

[a] S = standard finish. V = vibrated.

same forward speed as is customarily used. The number of passes was limited to three as in the former tests, this being considered the maximum allowable from the standpoint of production. However, it was found necessary to exceed two passes in only five cases.

With the set-up as described above, the vibrations could be distinctly felt by a person standing on the ground outside the line of forms. Attention, however, should be called to the fact that the usual standard-weight screeds were employed in this work. These screeds with attachments weigh approximately the same per foot of length for various widths of road. It is probable that a somewhat greater vibratory effect would have been secured had the machine been equipped with lighter screeds. A somewhat greater effect would probably have been obtained, even with the standard screeds, had an 18- or 20-ft. section been tested instead of a 9-ft. section.

## PROPORTIONS AND CONSISTENCY OF CONCRETE (SERIES A)

For each combination of fine and coarse aggregate, a so-called "base mix" was established, designed so that the combinations containing the coarse sand would have a cement factor of approximately 6.0 sacks per cu. yd. For the corresponding mixes with fine sand, the proportion of sand was reduced so as to compensate for the difference in grading without changing the water-cement ratio or the proportion of coarse aggregate. Preliminary laboratory tests indicated a reduction in sand content of one-tenth part in the case of gravel and two-tenths part in the case of stone and slag. This procedure resulted in the use of a slightly higher cement factor for the fine sand mixes, the maximum difference being 0.3 sack in the case of the stone concrete. Values of the ratio of $b$ to $b_0$ dry rodded ranged from 0.73 to 0.78, depending upon the materials being combined. Each of these base mixes was designed to produce concrete with a slump of about 2 in. and was used in a section constructed without vibration as a standard of comparison. For each standard section there were constructed five sections, in all of which the concrete was somewhat drier than the standard and all of which were vibrated. In two of the vibrated sections, the proportions were the same as the standard or base mix and the drier consistency was secured by lowering the water content until slumps approximately 1 in. and $\frac{1}{2}$ in. were obtained. In the other three, the water-cement ratio used in the standard or base mix was maintained constant and the drier consistency was secured by adding successively one-fourth, one-half and three-fourths part coarse aggregate by volume to the base proportion. A set of six sections, one screeded and five vibrated, formed one day's run. As previously noted, the proportions, slump, water-cement ratio and other data for each section comprising series A are shown in Table II. With the exception of the slag in combination with fine sand, two complete rounds were run, making a total of 66 sections, two with each aggregate combination.

## DISCUSSION OF RESULTS (SERIES A)

The data will be discussed first with the idea of showing in a broad general way the effect of vibration on the average of all comparable concretes containing the various aggregate combinations as compared with the base mix which was placed as the standard of comparison. Thus, referring to Table II, the results of tests on sections Nos. 2, 8, 14, 20, 38, 43, 50 and 56 have been averaged and will be compared to a similar average of the eight corresponding base sections (sections Nos. 1, 7, 13, 19, 37, etc.) to give a general

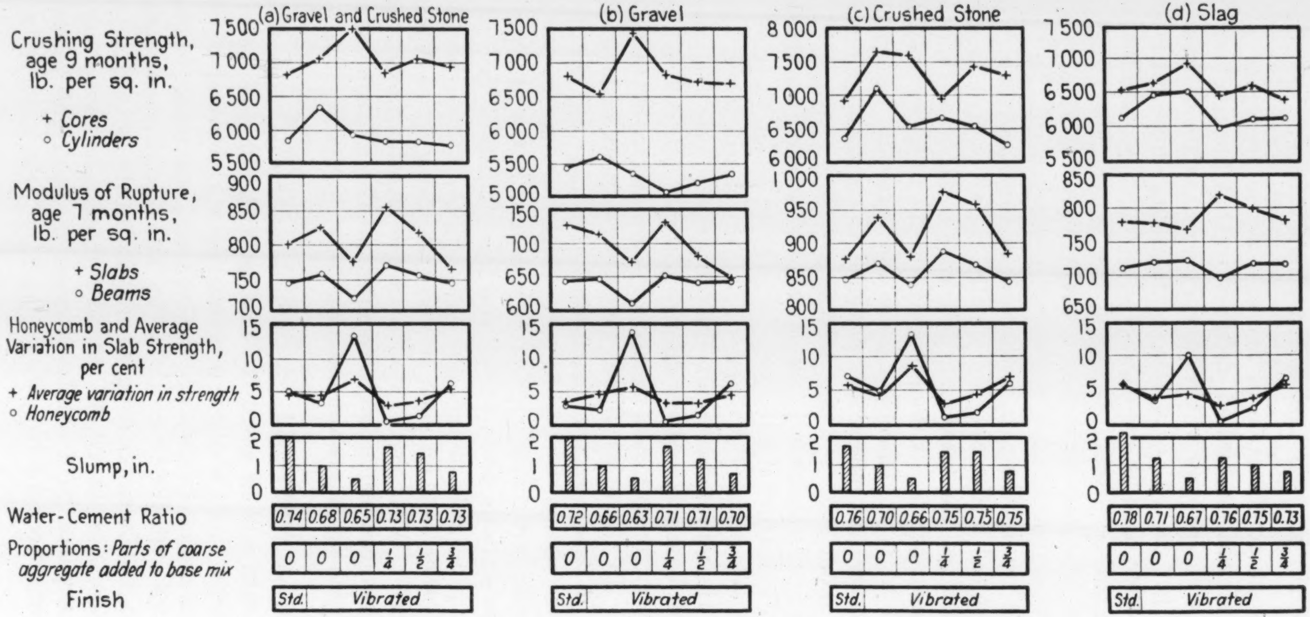

FIG. 4.—Average of Sections Containing Gravel, Crushed Stone and Slag (Series A).

picture of the effect of reducing the slump to about 1 in. without changing the proportions. In a similar way, the average results on sections Nos. 3, 9, 15, 21, etc., will be compared to the base mix and so on. It will be noted that each average value for slab strength obtained as above noted includes the results of two tests on each of four aggregate combinations; that is, gravel with coarse sand, gravel with fine sand, crushed stone with coarse sand and crushed stone with fine sand. Each value, therefore, represents the average of 32 individual tests. It is felt that these four combinations represent fairly well the typical aggregates in use at the present time. The results for slag are not included in this grouping; first, because slag is not a typical aggregate from the standpoint of distribution and, second, because in round 2 slag was not tested in combination with fine sand so that an average including slag could not be obtained from a series containing an equal number of tests of each combination.

A discussion of the data from the above standpoint will be followed by a comparison of the effect of variations in material type combinations; that is, the effect of vibration on gravel concrete *versus* crushed stone concrete, etc. This, in turn, will be followed by a detailed discussion of uniformity as revealed by a comparison of the strength and percentage of honeycomb in each of the four test slabs comprising each test section. It is felt that a study of the data from this standpoint is equally as important as a discussion of average results because of the fact that uniformity of quality may be considered as important a characteristic as average quality.

*Discussion of Average Results:*

The average values for the test results calculated as indicated above are shown in Fig. 4 (*a*).

An inspection of this chart reveals a number of interesting general relations. For instance, it will be noted that the vibrated slabs containing the base mix at 1-in. slump (values shown in the second vertical column of Fig. 4 (*a*)) show somewhat higher strengths and somewhat less honeycomb than the standard-finish base mix at 2-in. slump (column 1). In view of our experience in the first series, where the use of a consistency less than 2-in. slump when finished in the usual way resulted in lower slab strength and increased honeycomb, it seems reasonable to assume that the improvement in quality is due to the method of finishing. However, when we come to the vibrated base mix at ½-in. slump (column 3) we find a decrease in slab strength as well as a marked increase in honeycomb and less uniformity in strength. This would indicate obviously that the ½-in. slump is too dry for satisfactory placement, even with the use of vibrators as operated in these tests. The only increase in strength for this mix is noted in connection with the crushing strength of cores drilled from the sections. However, this lack of concordance between flexural and crushing strength is not surprising in view of the care taken in drilling cores to select portions of the slabs free from honeycomb. A badly honeycombed core would not be tested in ordinary work so it was deemed best to confine these tests to sound, homogeneous concrete in so far as possible and to study the effect of honeycomb through a study of variations in flexural strength as well as by direct measurement of the amount of honeycomb in each slab. With the exception of the sections in group 3 (½-in. slump) the spread between core and cylinder strength is just about as great for the standard finish sections as for the vibrated sections. This would indicate that, except in the case of the very dry mixes, the relatively higher core strengths are not due to the effect of vibration. In the case of the ½-in. slump concrete, it is probable that vibration did tend to increase the crushing strength of the concrete. On the other hand, the cylinders showed a decrease due to the fact that the ½-in. slump concrete was probably outside the range of consistency to which the laboratory water-cement ratio-strength relation applies.

In the three groups of sections in which the slump was decreased by increasing the proportion of coarse aggregate instead of reducing the water-cement ratio (columns 4, 5 and 6), it will be observed that the addition of coarse aggregate does not affect the crushing strength of the concrete, but that the flexural strength of the vibrated slabs is increased by the addition of one-fourth part coarse aggregate and is reduced as the amount of coarse aggregate is increased beyond one-fourth part. These trends seem entirely reasonable when we consider that the core strengths are not affected to the same extent as flexural strength by variations in the uniformity of the concrete in the slab. It is interesting to note that, both from the standpoint of average flexural strength, as well as uniformity in strength, the sections in group 4 containing one-fourth part additional coarse aggregate show the best results. It is true that the variations in strength and uniformity are not of considerable magnitude, the maximum difference in slab strength (column 4 as compared with column 6) being only about 12 per cent of the strength of the base mix. The various trends, however, are so consistent and appear so reasonable from a theoretical point of view that they may be assumed to be indicative of real variations in quality and so may be used as the basis for conclusions as to the effect of finishing on the results.

The values for each of the six material combinations from which the general averages were obtained have been regrouped in Figs. 4 (*b*) and (*c*) and 5 in order to ascertain to what extent variations in materials may affect the results. The results obtained with gravel as coarse aggregate are plotted in Fig. 4 (*b*), with similar data for crushed stone in Fig. 4 (*c*). It will be observed that for similar mixes and methods of finishing the sections containing crushed stone as coarse aggregate show somewhat higher crushing strengths and considerably higher flexural strengths than the sections containing gravel. Attention is called to the fact that these differences are due to variations in the physical properties of the particular aggregates used in these tests and not merely to the fact that one aggregate happens to be crushed stone and the other gravel. Aggregates from other sources might well have shown entirely different relative strengths both in compression and in flexure. In Fig. 5 the average results of tests on concrete containing gravel and crushed stone in

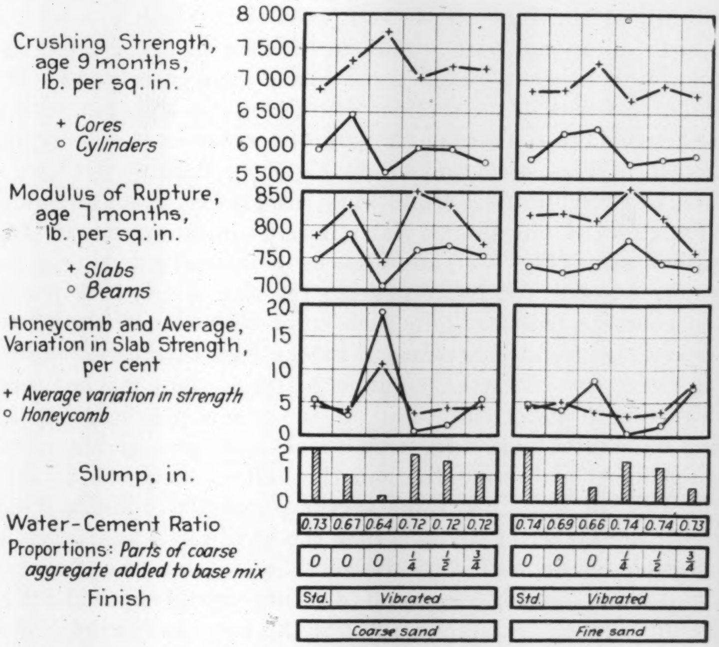

FIG. 5.—Average of Sections Containing Gravel and Crushed Stone (Series A).

combination with coarse sand are compared with the corresponding averages for concrete containing fine sand. The results obtained with slag, which were not included in the general average, are shown in Fig. 4 (*d*).

An inspection of each chart reveals, in general, the same trends as are shown in Fig. 4 (*a*). In all cases the highest slab strength as well as the least honeycomb is found in the vibrated group containing one-fourth part additional coarse aggregate (column 4 in each). Likewise, increasing the quantity of coarse aggregate beyond one-fourth part in all cases decreased the slab strength as well as the uniformity of the concrete as compared with results secured with one-fourth additional part. The curves showing these relations are remarkably consistent for all three coarse aggregate types. It will be noted also that in the sections containing crushed stone and slag, even the concrete containing the largest amount of coarse aggregate (column 6 of Figs. 4 (*c*) and (*d*)) is higher in flexural strength than the base mix unvibrated. Uniformity, also, is just about as good.

The effect of vibration on slab strength is most marked in the case of crushed stone (Fig. 4 (*c*)) and least in the case of gravel (Fig. 4 (*b*)). This may be observed by comparing the slab strength of the unvibrated concrete (column 1 in each case) with the strengths of the corresponding vibrated sections. In the case of crushed stone, all but the very dry concretes (columns 3 and 6) show marked increase in slab strength under vibration. In the case of gravel, group 4 containing one-fourth part additional coarse aggregate, is the only one showing higher slab strength than the base or unvibrated concrete. In the case of slag, the three sections carrying additional coarse aggregate are higher in strength than the unvibrated base mix. Both the crushed stone and slag concretes containing the drier mixes (columns 3 and 6 of Figs. 4 (*c*) and (*d*)) show slab strength about equal to the unvibrated concrete in spite of the fact that group 3 in each case has considerably more honeycomb than the base mix. It is interesting to note that, in general, the uniformity of the slag concrete is as good as that shown for crushed stone and gravel.

The inability of the vibrators to finish very dry concrete ($\frac{1}{2}$-in. slump) is shown by the relatively low strengths and high percentages of honeycomb in the slabs comprising the third group in each case (column 3 of Figs. 4 (*b*), (*c*) and (*d*)). Variation in consistency apparently had a smaller effect on slab strength of slag concrete than either of the other coarse aggregates. Somewhat less honeycomb in the case of the dry mix (group 3) may also be noted. The marked difference in crushing strength of gravel concrete cylinders with respect to cores as compared with similar results with crushed stone and slag should also be noted. In the former case the cores average about 1500 lb. per sq. in. higher than the cylinders, which is a considerably greater difference than is shown for either the stone or slag concrete.

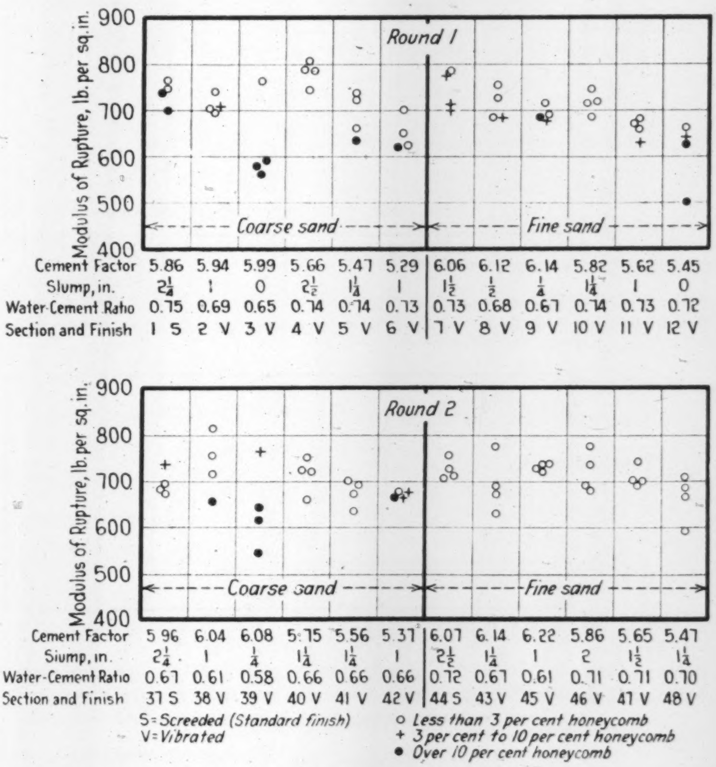

FIG. 6.—Flexural Strength of Gravel Concrete Pavement Slabs, Age 7 months (Series A).

*Coarse Versus Fine Sand:*

In Fig. 5, the test results for all sections using crushed stone and gravel in combination with coarse sand as fine aggregate have been averaged and are compared with the results for the corresponding concretes in which the fine sand was used.

The same general relationships appear except that the adverse effect of using the dry mix is more marked in the case of coarse sand. The average slump is, however, considerably lower; that is, ⅛ in.

as compared with ½ in. for the fine sand. The average difference in slab strength as compared with beam strength is also more marked in the case of the fine sand, especially for the unvibrated concrete and for the two groups of vibrated concrete containing the base mix with lower water-cement ratio. The tendency for the slab strengths to drop at a greater rate than the beam strengths for increasing percentages of coarse aggregate should also be noted in this as well as in the other graphs. The general agreement between the results of strength tests on cores, cylinders and beams for concretes having practically the same water-cement ratio (columns 1, 4, 5 and 6) is also of interest.

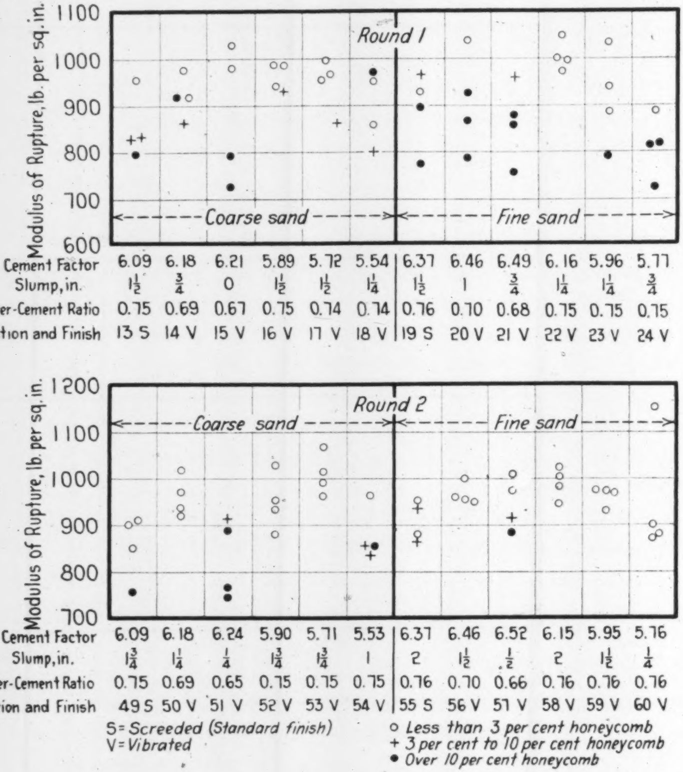

FIG. 7.—Flexural Strength of Stone Concrete Pavement Slabs, Age 7 months (Series A).

*Comparison of Uniformity of Individual Sections:*

In order to bring out clearly the effect of honeycomb on slab strength, the results of the tests for flexural strength of each of the four 27-in. slabs in each test section have been plotted in Figs. 6, 7 and 8. In practically every case we find that the highest strengths obtained for any test section were on slabs containing less than 3 per cent honeycomb, whereas those slabs showing an excess of 10 per cent honeycomb developed much lower strengths. The effect of using dry consistencies on the extent of honeycomb is also brought out. Marked examples of the effect of honeycomb on strength and the effect of consistency on the amount of honeycombing are sections 3, 12 and 39 in Fig. 6 (all of which showed ¼ in. or less slump), sections 15, 21, 24 and 51 of Fig. 7 (all ¾ in. or less slump) and section 27 in Fig. 8. On the other hand, those sections showing the least honeycomb and greatest uniformity are, in general, the vibrated sections in which the slump was in excess of 1 in. There are some

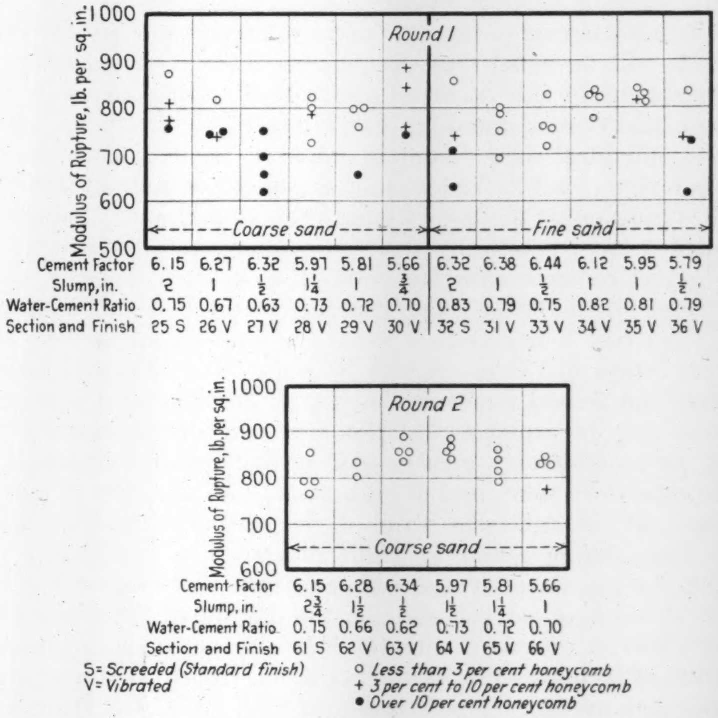

FIG. 8.—Flexural Strength of Slag Concrete Pavement Slabs, Age 7 months (Series A).

exceptions to this, as for instance, section 45 (Fig. 6) with 1-in. slump and less than 3 per cent honeycomb in any slab. This section also showed a high degree of uniformity. It will be noted that honeycombing exists in almost all of the standard finish sections for an average slump of $2\frac{1}{4}$ in. or less. Section 44 (Fig. 6) with $2\frac{1}{2}$-in. slump, and section 61 (Fig. 8) with $2\frac{3}{4}$-in. slump, were the only ones with all slabs showing less than 3 per cent honeycomb.

Further reference to Figs. 6, 7 and 8 will reveal another interesting fact. It will be noted that, in general, there is considerably less honeycombing in the sections constructed during round 2 than in those built during round 1. For instance, 40, or 28 per cent of the 144 individual slabs tested in round 1, showed more than 10 per cent honeycomb, whereas only 11, or 9 per cent of 118 slabs tested during round 2, showed more than 10 per cent. Two groups in round 2 were practically free from honeycomb exceeding 3 per cent, gravel concrete with fine sand (Fig. 6) and slag with coarse sand (Fig. 8), the latter group containing only one slab with more than 3 per cent honeycomb, whereas the former contained no slabs showing honeycomb in excess of 3 per cent. The difference between the first and second round in the case of the fine sand and gravel concrete may be explained by the fact that during round 2 the slump was considerably greater than during round 1 although the same proportions were used in both cases. As a matter of fact the average net water-cement ratio during round 2 was somewhat lower than during round 1. This difference in consistency may possibly be explained by the comparatively low relative humidity of the atmosphere (48 per cent) on the day on which sections 7 to 12 were laid as compared with the humidity noted during the construction of sections 43 to 48 (65 per cent). Both days were sunny and hot with average temperatures over 90 F. (30 C.). There seems to be no other reason to account for the marked difference in consistency which was observed.

This difference in consistency illustrates very forcibly the difficulty of controlling this very essential property of concrete under field conditions. Here we have a condition where variations in the humidity undoubtedly affected the consistency to a marked degree. Under such conditions, it is impossible to maintain both proportions and water-cement ratio and to expect the consistency to also remain constant. Therefore, inasmuch as consistency must be closely controlled, and it is impractical to be constantly changing batch weights in order to take care of variations therein, the question arises as to whether we should attempt to construct concrete pavements under a straight water-cement ratio specification, rather than a specification in which the proportions and consistency are fixed and the water content is allowed to vary up to a fixed maximum.

In the case of the coarse sand and slag combination (Fig. 8) the marked difference in honeycombing observed between the two rounds is not explained by differences in consistency. Only in the case of the unvibrated concrete was the difference in slump more than ½ in. Sections 27 and 63 show the greatest divergence so far as workability is concerned. The slump in both cases was ½ in. Sections 25 to 30 (round 1) were laid on a damp, cloudy day (average temperature 76 F. (24 C.), relative humidity 88 per cent) whereas sections 61 to 66 were laid on a rather cool clear day with average temperature of 69 F. (20 C.) and relative humidity of 46 per cent. Under such conditions, it would be reasonable to suppose that the concrete laid during round 1 when the relative humidity was 88

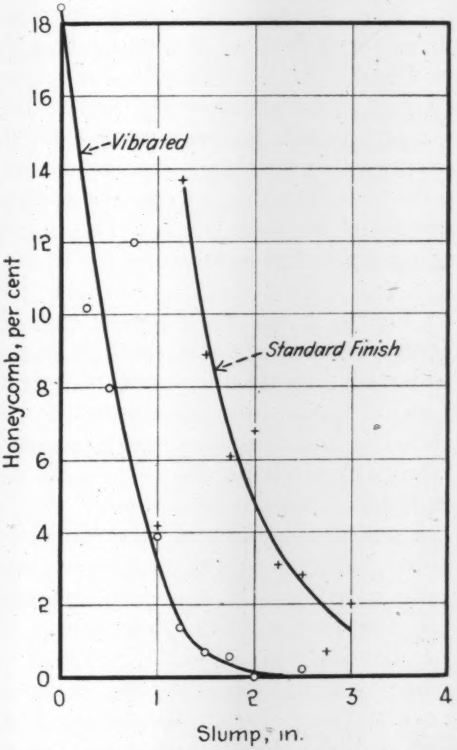

FIG. 9.—Relation Between Honeycomb and Slump.

per cent would not dry out so fast as the concrete laid during round 2 when the atmosphere was much drier. Actually, the reverse appeared to be the case.

*Relation Between Workability and Slump:*

To show the average effect of variation in consistency (slump) on the workability of both the vibrated and unvibrated concrete, the average percentage of honeycomb in all slabs constructed of concrete having a given consistency and finished in a given manner has been determined for each value of slump within the range studied, that is, between 0 and 3 in. The results are shown graphically in Fig. 9. In this figure, the curve showing the relation between slump and percentage of honeycomb for each method of finishing has been drawn so as to represent a weighted average of the various points. In other words, due consideration was given to the number of individual tests represented by each point. This number varied from 1 to 14, depending upon the number of sections having a given slump.

Referring to Fig. 9, it will be observed that, for a given consistency, the average amount of honeycomb in the slabs finished by vibration is considerably less than in the slabs finished by the standard method. It would appear from these data that the 2-in. minimum slump recommended in the previous report for paving mixes to be finished under standard methods did not, under the conditions of these tests, completely eliminate honeycomb. As a matter of fact, at 2-in. slump an average amount of honeycomb of approximately 5 per cent is noted, whereas in the first series honeycombing was almost entirely eliminated at 2-in. slump, except where the so-called gravel, grading B, was used as coarse aggregate. (See Fig. 13, *Proceedings*, Am. Soc. Testing Mats., Vol. 31, p. 476 (1931).)

Several explanations may be advanced to account for this difference. In the first place the coarse aggregate used in the first series was divided in three sizes, whereas only two sizes were used in the present series. This may possibly account for the difference in workability. Again, in the original series the subgrade was protected with tar paper whereas, in the present series, the slabs were cast direct upon the subgrade. It is possible that the tar paper, acting somewhat as a form, prevented the segregation of mortar and coarse aggregate on the bottom surface of the slab, thus reducing the percentage of visible honeycomb somewhat.

Of course, from the standpoint of this investigation, the most important consideration is the difference in slump for standard finished concrete as compared with vibrated concrete of the same degree of workability (percentage of honeycomb). This difference seems to be in the neighborhood of 1½ in. For instance, in order to insure concrete substantially free from honeycomb (and for the purpose of this discussion 3 per cent will be considered the upper limit for this condition) a minimum slump of about 2½ in. will be required when the concrete is finished in the usual way compared with about a 1-in. slump when the concrete is vibrated. It will be seen therefore that, if anything, the minimum slump of 2 in. recommended in the first report instead of being higher than necessary was not high enough to eliminate honeycombing. In other words, the results of the present series not only substantiate the conclusions reached in the first report, but seem to indicate that a minimum slump of 2½ in. instead of 2 in. may be advisable in cases where the concrete is finished in the usual way.

A study of the distribution of honeycomb on the bottom of the four 27-in. slabs composing each test section of series A reveals some interesting facts. For the purpose of this discussion the four slabs in a test section will be numbered from 1 to 4, beginning on the right-hand side when facing in the direction of the movement of the paver. Two 27-cu. ft. batches of concrete were required to construct each section. The first batch was dumped from a position in the center of slab No. 3 and spread both ways into slabs Nos. 2 and 4. The second batch was dumped from over the center of slab No. 1 and spread out into slab No. 2, overlapping the concrete from the first batch. The longitudinal valley between adjacent batches was therefore in slab No. 2. It may be noted that of the 11 standard finished sections in series A, slab No. 2 showed the greatest amount of honeycomb in 9 cases, with slab No. 1 showing the maximum in one case and slab No. 4 the maximum in another. This would indicate a distinct tendency to honeycomb in the valley between the two batches and points to the necessity for care in spotting batches of concrete during actual construction in order to minimize this condition.

Of the 43 vibrated sections showing honeycomb, slab No. 2 contained the largest amount in 10 cases, slab No. 4 in 22 cases, and slab No. 1 in 11 cases. This distribution shows the greatest amount of honeycomb in the end slabs (Nos. 1 and 4) in about 75 per cent of the cases and is quite different from that occurring with the screeded sections, where 82 per cent of the sections showed maximum honeycomb in slab No. 2. Inasmuch as the procedure used in dumping was exactly the same, an explanation of the difference may lie in the vibratory effect which was possibly somewhat greater in the center of the screed than at the ends.

*Honeycomb at Separators:*

At the time of the former investigation, a question was raised as to the effect of the wooden separators installed for the purpose of creating planes of weakness on the amount and distribution of honeycomb in the slabs. It was felt that the separators might have interfered with the distribution of concrete on the subgrade resulting in the concentration of honeycomb areas at and near the separators. A very careful inspection of all of the test slabs comprising this series has failed to reveal any particular trend along this line and it is felt that there was no tendency in this direction. In other words, it seems safe to assume that, other things being equal, the same amount and distribution of honeycomb would have occurred on an actual pavement as was observed in this test.

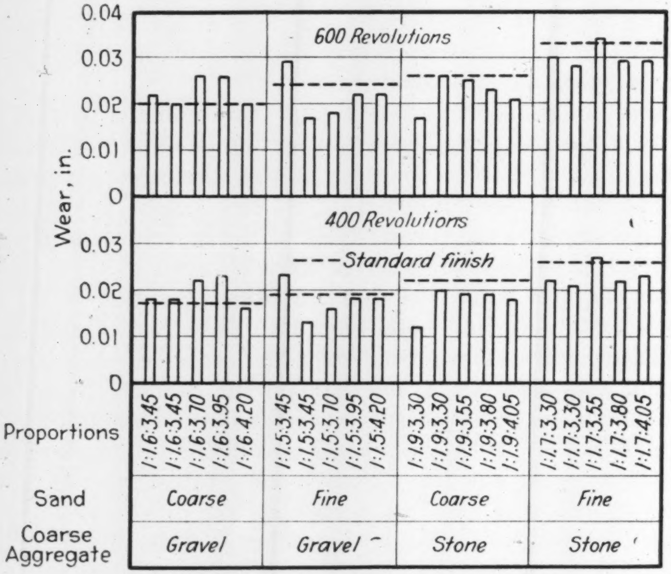

FIG. 10.—Effect of Vibration on Surface Wear.

*Surface Wear:*

The results of the tests for surface wear are shown in Fig. 10. The depth of wear is reported at the end of both 400 and 600 revolutions. Each value is the average of either two or three individual tests on different slabs. For each group of six, the average hardness of the surface of the five vibrated sections may be compared directly with the standard finish section for that particular group. In general, the vibrated sections show slightly less surface wear, although the difference is not sufficiently marked to warrant any general conclusion regarding this property of the concrete. It may safely be assumed, however, on the basis of these tests that vibration does not adversely affect the surface hardness of the pavement.

It is of interest to note that the hardness of the standard finish concrete appears to be slightly affected by the material combinations. Sections containing the fine sand seem to be somewhat lower in resistance to wear than the corresponding sections with coarse sand. The same comment applies to the crushed-stone concrete as compared with gravel. In the latter case, the difference in hardness is probably due to the fact that a somewhat higher water-cement ratio was used in the crushed-stone concrete. Inasmuch as wear was confined to the mortar surface, the type of coarse aggregate did not, of course, directly affect the results.

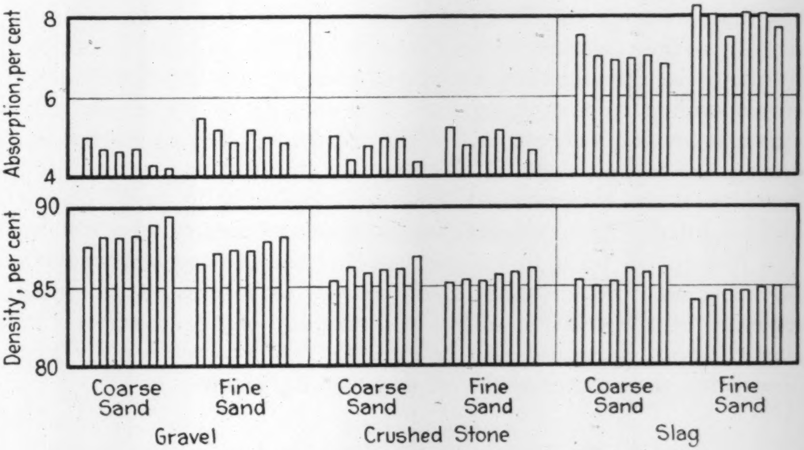

FIG. 11.—Density and Absorption Tests (Series A).

NOTE.—Sections in each group arranged from left to right as in Fig. 4.

*Density and Absorption:*

In Fig. 11, there have been plotted average values for density and absorption of cores drilled from the sections in series A. It will be seen that in each group the final densities of the vibrated concretes are, with the exception of groups 2 and 3, slag - coarse sand combination, all higher than the densities of the corresponding base mixes and that, in general, the density increases with increased coarse aggregate content (Nos. 4, 5 and 6 in each group). These variations in density could, of course, be accounted for on the basis of the decreased water or increased proportion of coarse aggregate in the mix provided it could be assumed that the freshly mixed concretes were, in all cases, free from air voids. However, it is felt in the case of series A, where all of the vibrated sections were drier than the base mix and where several carried more coarse aggregate, that this assumption cannot be made. In other words, it seems reasonable to assume that, in the case of the vibrated sections, all of the freshly mixed concrete contained more air voids than the base mix. The fact that the final densities arrange themselves in about the same relative order as the densities of the freshly mixed concretes calculated without correction for air voids is an indication that air voids were eliminated during the finishing operation. Of course, the important feature, so far as these tests are concerned, is that we actually obtained, by the use of the vibratory method of finishing, the increased densities corresponding to the changes in the quantity of water and coarse aggregate which were made. Although density tests were not made during the former tests, it is extremely improbable, in view of the inability of the conventional finishing machine to properly place the harsher mixes, that corresponding increases in density were obtained.

Absorption tests, in general, parallel the density tests, as would be expected. Absorption is, of course, of interest as a possible measure of the relative ultimate durability of the concrete. The slag concrete, as would also be expected, shows considerably higher absorption than either of the other coarse aggregates.

## SERIES B—TESTS TO DETERMINE EFFECT OF DELAYED FINISHING (JOHNSON METHOD)

In series B, a method of finishing concrete pavement slabs proposed by T. H. Johnson of Sioux City, Iowa, and known as the "Johnson method" was investigated.[1] In all, 40 sections were constructed, 16 with gravel as coarse aggregate, 16 with crushed stone as coarse aggregate, and 8 in which the total aggregate was a sand-gravel mixture approximating in grading the so-called "Platte River gravel" used extensively in Nebraska and western Iowa. The gravel and crushed stone sections were further subdivided as to fine aggregate, an equal number of sections in each group being constructed with coarse and fine sand. Aggregate gradings and other characteristics are given in Table I.

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[1] A detailed discussion of the theory underlying this method was presented by T. H. Johnson at the Twenty-third Annual Meeting of the American Concrete Institute in 1927 (see *Proceedings*, Am. Concrete Inst., Vol. 23, p. 458).

TABLE III.—DATA ON MIXES, SERIES B.

| Section[a] | Proportions by Volume, Dry-Rodded | Cement Factor, sacks per cu. yd. | Water-Cement Ratio, Corrected | Ratio, $\frac{b}{b_o}$ | Slump, in. |
|---|---|---|---|---|---|
| COARSE SAND AND GRAVEL (ROUND 1) | | | | | |
| No. 67 S | 1:1.6:3.45 | 6.0 | 0.67 | 0.76 | 1¾ |
| No. 68 J | 1:1.6:3.45 | 5.9 | 0.71 | 0.76 | 2½ |
| No. 69 S | 1:2.1:4.45 | 4.8 | 0.76 | 0.79 | 1½ |
| No. 70 J | 1:2.1:4.45 | 4.8 | 0.82 | 0.79 | 2½ |
| FINE SAND AND GRAVEL (ROUND 1) | | | | | |
| No. 71 S | 1:1.5:3.45 | 6.1 | 0.69 | 0.78 | 1¼ |
| No. 72 J | 1:1.5:3.45 | 6.0 | 0.76 | 0.77 | 2¾ |
| No. 73 S | 1:2.0:4.45 | 4.8 | 0.88 | 0.80 | 1 |
| No. 74 J | 1:2.0:4.45 | 4.8 | 0.95 | 0.79 | 2 |
| COARSE SAND AND CRUSHED STONE (ROUND 1) | | | | | |
| No. 75 S | 1:1.9:3.3 | 6.1 | 0.75 | 0.75 | 2½ |
| No. 76 J | 1:1.9:3.3 | 6.1 | 0.77 | 0.74 | 2¾ |
| No. 77 S | 1:2.4:4.3 | 5.0 | 0.87 | 0.79 | 1½ |
| No. 78 J | 1:2.4:4.3 | 4.9 | 0.95 | 0.78 | 3 |
| FINE SAND AND CRUSHED STONE (ROUND 1) | | | | | |
| No. 79 S | 1:1.7:3.3 | 6.4 | 0.76 | 0.78 | 2½ |
| No. 80 J | 1:1.7:3.3 | 6.3 | 0.80 | 0.77 | 3½ |
| No. 81 S | 1:2.2:4.3 | 5.1 | 0.96 | 0.81 | 2 |
| No. 82 J | 1:2.2:4.3 | 5.1 | 0.96 | 0.81 | 2¼ |
| COARSE SAND AND GRAVEL (ROUND 2) | | | | | |
| No. 83 S | 1:1.6:3.45 | 6.0 | 0.67 | 0.76 | 2 |
| No. 84 J | 1:1.6:3.45 | 5.9 | 0.71 | 0.76 | 3 |
| No. 85 S | 1:2.1:4.45 | 4.8 | 0.79 | 0.79 | 2¾ |
| No. 86 J | 1:2.1:4.45 | 4.8 | 0.82 | 0.79 | 3½ |
| FINE SAND AND GRAVEL (ROUND 2) | | | | | |
| No. 87 S | 1:1.5:3.45 | 6.1 | 0.72 | 0.77 | 3 |
| No. 88 J | 1:1.5:3.45 | 6.1 | 0.72 | 0.77 | 2¾ |
| No. 89 S | 1:2.0:4.45 | 4.8 | 0.87 | 0.80 | 2¼ |
| No. 90 J | 1:2.0:4.45 | 4.8 | 0.90 | 0.79 | 3 |
| COARSE SAND AND CRUSHED STONE (ROUND 2) | | | | | |
| No. 91 S | 1:1.9:3.3 | 6.1 | 0.77 | 0.74 | 2 |
| No. 92 J | 1:1.9:3.3 | 6.0 | 0.80 | 0.74 | 3¾ |
| No. 93 S | 1:2.4:4.3 | 4.9 | 0.97 | 0.77 | 2½ |
| No. 94 J | 1:2.4:4.3 | 4.8 | 0.99 | 0.77 | 3¾ |
| FINE SAND AND CRUSHED STONE (ROUND 2) | | | | | |
| No. 95 S | 1:1.7:3.3 | 6.4 | 0.77 | 0.78 | 2 |
| No. 96 J | 1:1.7:3.3 | 6.3 | 0.79 | 0.77 | 3 |
| No. 97 S | 1:2.2:4.3 | 5.1 | 0.95 | 0.81 | 2 |
| No. 98 J | 1:2.2:4.3 | 5.0 | 0.98 | 0.80 | 3 |
| PLATTE RIVER GRAVEL (ROUND 1) | | | | | |
| No. 99 S | 1:4 | 6.4 | 0.93 | .... | 1½ |
| No. 103 J | 1:4 | 6.3 | 1.03 | .... | 2½ |
| No. 101 S | 1:4.5 | 5.8 | 1.05 | .... | 2 |
| No. 105 J | 1:4.5 | 5.7 | 1.10 | .... | 2 |
| PLATTE RIVER GRAVEL (ROUND 2) | | | | | |
| No. 100 S | 1:4 | 6.4 | 0.97 | .... | 1¾ |
| No. 104 J | 1:4 | 6.3 | 1.00 | .... | 2 |
| No. 102 S | 1:4.5 | 5.8 | 1.05 | .... | 1½ |
| No. 106 J | 1:4.5 | 5.7 | 1.10 | .... | 2¼ |

[a] S=standard finish. J=Johnson method.

## PROPORTIONS (SERIES B)

For each aggregate combination, except the Platte River gravel, a base mix of the same proportions as used in series A was set up as the standard of comparison. This base mix was finished with the standard double-screed finishing machine in the usual way. For direct comparison with this standard or base mix there were constructed three sections with mixes varying from the base as indicated below. This made a total of four sections per round for each aggregate combination. For the second section of each group the base proportion was used but the water content was increased to give a slump of approximately 3 in. instead of 2 in. This section was finished by the Johnson method. The third section in each group was proportioned to contain one-half part more fine aggregate and one part more coarse aggregate by volume than the base mix and was finished in the standard manner. The water content in this section was adjusted to give a slump of about 2 in. and the water-cement ratio averaged about 0.15 higher than the base mix. The cement factor averaged 1.2 bags per cu. yd. less than the base. The fourth section was a mix similar to the third except that the water content was increased to give approximately 1 in. greater slump. This section was finished by the Johnson method in the same manner as the second section in the group. In Table III the proportions, slump, water-cement ratio and method of finishing for each of the sections in series B are given.

It will be seen that this arrangement makes it possible to compare for each group of four sections the results secured by the Johnson method with the standard, first, on the basis of the same mix except for a somewhat wetter consistency and, second, on the basis of not only a wetter but also a considerably leaner mix than the base. It is also possible to compare the lean mix Johnson-finished sections with a similar mix containing less water and finished in the usual way.

The data for the eight sections in which the sand-gravel mix was used are also shown in Table III. Here the base mix, standard finish, was 1:4 by volume, with sufficient water to give approximately a 2-in. slump. As a comparison, a similar mix with more water was finished by the Johnson method. The group of four was completed by two sections, 1:4½ mix by volume, one finished by the standard method, and one, somewhat wetter, by the Johnson method. Two complete rounds of tests, for each aggregate combination, were run in series B, making the total of 40 sections in groups of four each as above indicated. The laying of these sections was started on September 29, 1931, and completed on October 5, 1931, progress being at the rate of 8 sections per day.

## METHODS OF FINISHING (SERIES B)

The procedure used in finishing the concrete by the Johnson method was as follows:

Immediately after the concrete was deposited on the subgrade it was struck off with a hand screed. It was then floated with a long-handled wooden float 12 in. wide by 20 in. long by $2\frac{1}{4}$ in. thick. The thickness was obtained through the use of a $1\frac{3}{4}$ in. cypress plank with a $\frac{1}{2}$-in. oak plank nailed to the bottom, the oak making contact with the concrete. After floating, the surface was rolled with a light-weight sheet-metal roller. The roller was operated from side to side of the pavement and was advanced about one foot longitudinally with each passage. The entire surface was rolled three times, after which it was belted. This was followed by the application of the so-called "dry mix" to the surface. This mixture was made up of one part cement to one part of fine sand by volume and was spread over the surface with shovels at a rate of one sack of cement per 20 sq. yd. of pavement. For a 7-in. pavement this would be equivalent to increasing the total amount of cement used about 0.25 bag per cu. yd. of concrete. Just enough of the mixture was applied to give a very thin continuous cover. The sand used for this purpose was the same as the fine sand used in the concrete mixture and contained approximately 5 per cent of moisture when mixed with the cement. After an interval of 11 to 50 min., depending upon the amount of water brought to the surface by the dry mix, the surface was again belted. This was followed by floating with

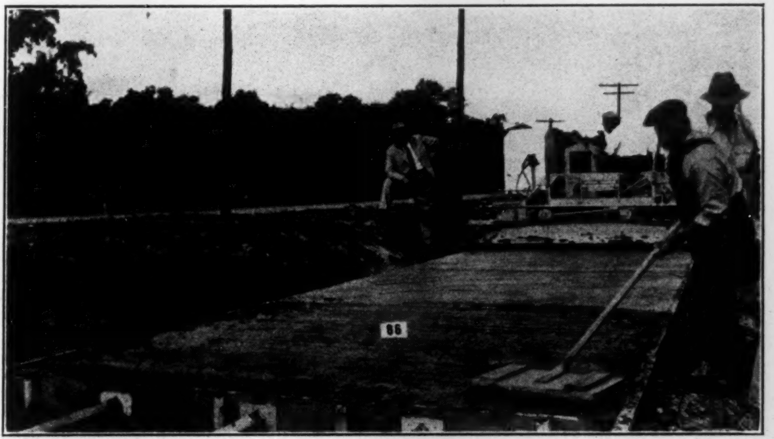

FIG. 12.—Floating with Long-Handled Wood Float, Finishing by the Johnson Method.

hand floats at intervals until there was little moisture brought to the surface and the concrete became sticky and was drawn along by the float. This general procedure was sometimes varied by belting between floating operations and by additional rollings. The number of floatings necessary to bring the surface to the required condition after the dry mix was applied varied from three to seven and the elapsed time from the placing of the concrete to the final floating varied from $2\frac{1}{4}$ to $3\frac{3}{4}$ hr.

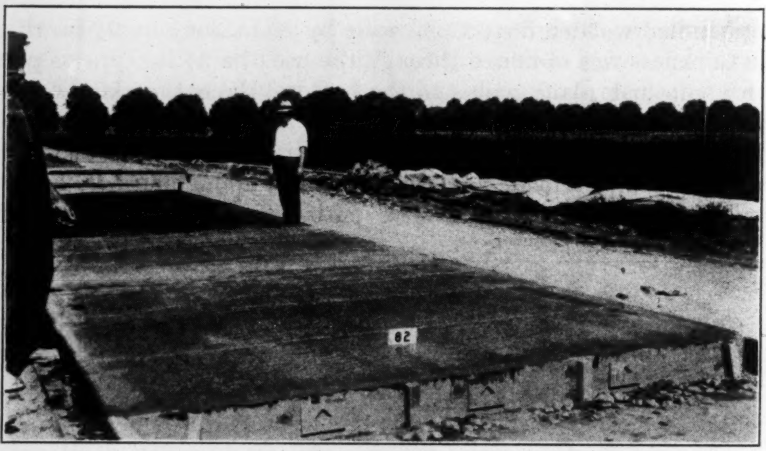

FIG. 13.—Surface Just After Belting and Before Application of Dry Mix, Finished by the Johnson Method.

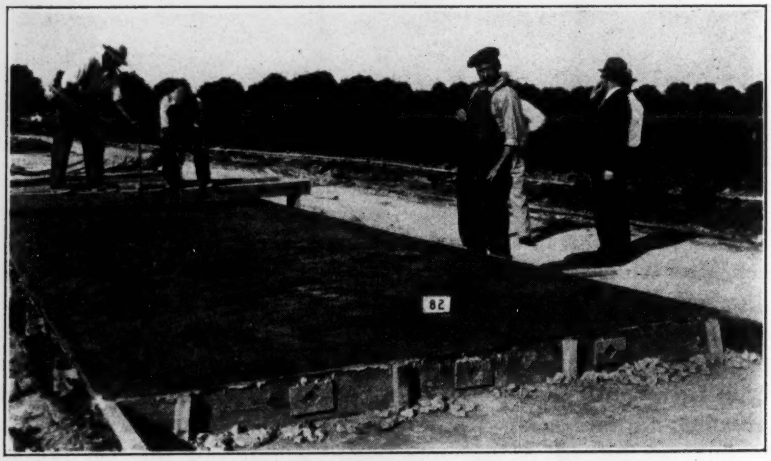

FIG. 14.—Finished Surface by the Johnson Method.

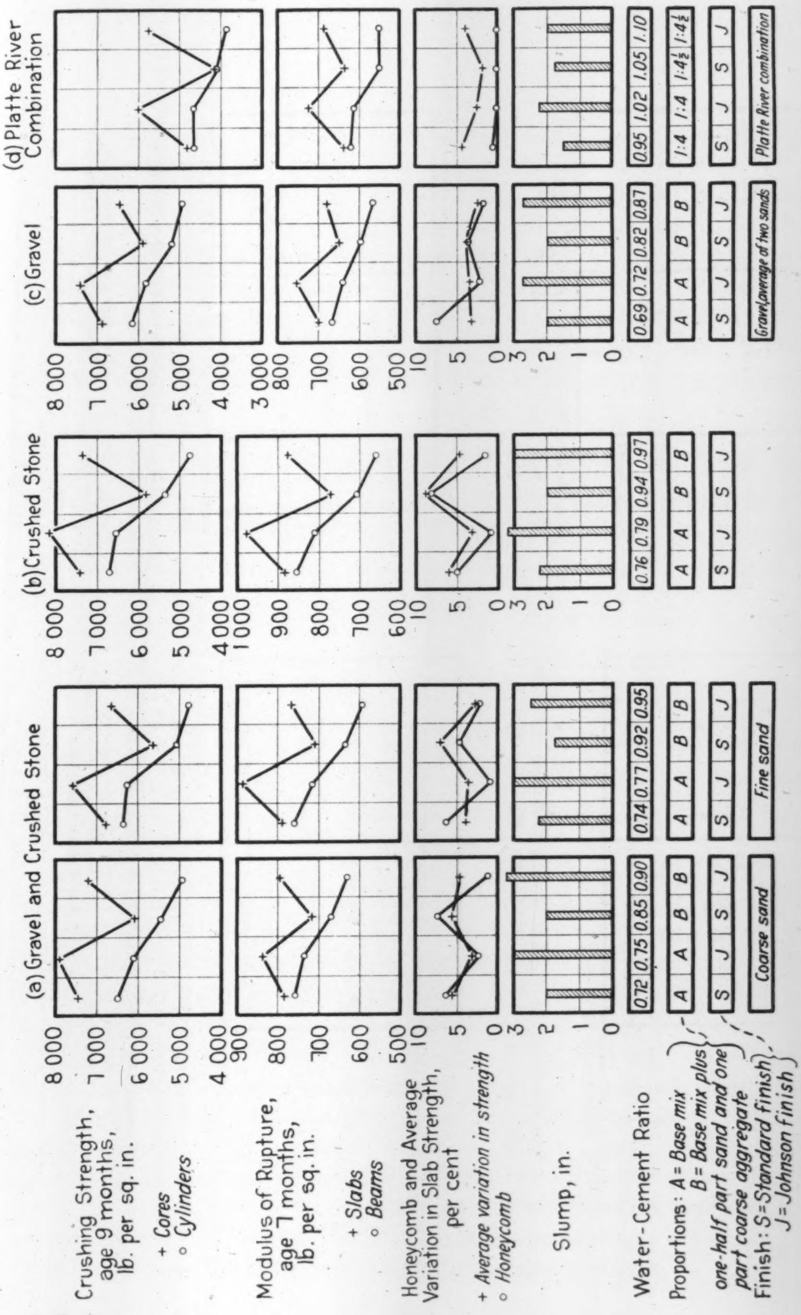

FIG. 15.—Average of Sections Containing Gravel, Crushed Stone and Platte River Combination (Series B).

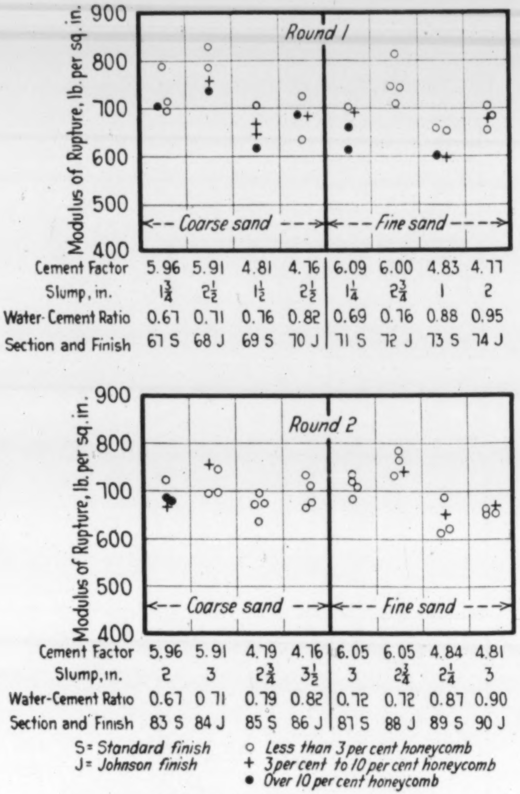

FIG. 16.—Flexural Strength of Gravel Concrete Pavement Slabs, Age 7 months (Series B).

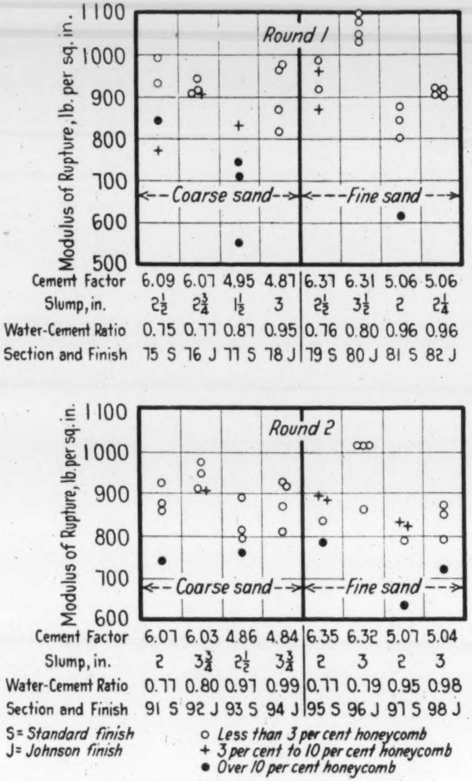

FIG. 17.—Flexural Strength of Stone Concrete Pavement Slabs, Age 7 months (Series B).

Various details of the operation as described above are illustrated in Figs. 12 to 14.

## DISCUSSION OF RESULTS (SERIES B)

The effect of the Johnson method of finishing on the strength and uniformity of the concrete is shown graphically in Figs. 15 to 18, inclusive. The data are shown in the same manner as the corresponding data for series A, and include average results of crushing strength tests on cores and cylinders, tests for modulus of rupture on slabs and control beams and determinations of uniformity, measured by the amount of honeycomb in the slabs and the average per cent variation in slab strength. In Fig. 15 (*a*), the average results of tests for the crushed-stone and the gravel concrete for each sand are shown. Figure 15 (*b*) gives similar data for the crushed stone concrete only, while in Fig. 15 (*c*) the corresponding results for the gravel and Platte River combination are shown. In the case of the crushed-stone and gravel concrete (Figs. 15 (*b*) and (*c*)) the values for the sections containing the fine and coarse sand have been averaged. Figures 16, 17 and 18 give the results of tests on individual slabs taken from the test sections.

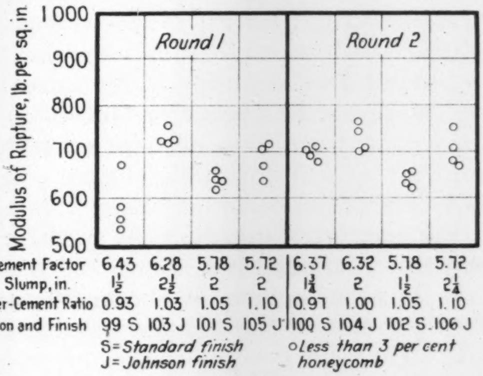

FIG. 18.—Flexural Strength of Platte River Combination Pavement Slabs, Age 7 months (Series B).

It will be noted that in the case of Fig. 15 the results are plotted so as to provide, for each proportion, a direct comparison between the strengths secured under the Johnson method and the standard method. Each point represents the average of tests on four sections, except that in the case of the Platte River concrete (Fig. 15 (*c*), right-hand panel) each point averages only two sections.

It will be noted that, although a somewhat wetter mix and consequently higher water-cement ratio was used in each Johnson-finished section as compared with the corresponding standard-finished concrete, in every case a higher core and slab strength was obtained on the sections finished by the Johnson method. On the other hand, the strengths of the control specimens representing the concrete going into the Johnson-finished slabs were lower than the corresponding standard sections. This, of course, would be expected because of the higher water-cement ratios. The higher strengths obtained for the Johnson finish are undoubtedly due, at least in so far as crushing strength is concerned, to the special manipulation which was given the concrete, resulting in the removal of considerable water before final consolidation took place. The control specimens were, of course, not subjected to this manipulation and consequently retained the excess water, resulting in a lowering of the strength. As to the increased slab strength shown by the Johnson-finished concrete, it is possible that a portion of the increase is due to the influence of consistency on honeycombing and, consequently, on slab strength, entirely apart from the removal of water during the finishing operation.

Referring to Fig. 15 (*a*), let us compare first the results of the core tests for the base mix (proportion A). The average crushing strength of sections containing both coarse and fine sand shows an increase for the Johnson sections of about 600 lb. per sq. in. or approximately 8 per cent of the average strength of the standard-finish base mix. The corresponding control cylinders show a slight decrease in strength, as would be expected. This decrease averages about 250 lb. per sq. in. and corresponds to an increase in water-cement ratio of 0.03. For the leaner mix (proportion B), the average increase in core strength is about 1000 lb. per sq. in., or approximately 18 per cent. The grand average increase, including all sections, is, therefore, about 13 per cent in favor of the Johnson method of finishing. Inasmuch as the only difference, aside from water content and consistency, was the method of finishing, it may be concluded that the higher average strength shown for the cores drilled from the Johnson sections is due to the method of finishing. It is also interesting to note that the average core strength of the Johnson-finished slabs in the lean mix (average water-cement ratio, 0.93) is only about 200 lb. per sq. in. lower than the corresponding cores for the richer-mix (average water-cement ratio, 0.73) standard finish. The cylinders are about 1600 lb. per sq. in. lower in strength.

The same general trends follow in the case of flexure. For proportion A the average increase in modulus of rupture in favor of the Johnson finish is about 75 lb. per sq. in. or roughly 9 per cent. For proportion B, the increase is about the same. The control beams, which were, of course, fabricated in the usual way, show no increase but, on the other hand, a decrease in strength with increase in water content in the same manner as the control cylinders. Considering the small number of specimens represented by each point (8 in the case of cores, cylinders and beams, and 16 in the case of slabs) the results are remarkably concordant. As in the case of the cores, the Johnson-finished slabs containing the lean mix show an average modulus of rupture only very slightly lower (about 10 lb. per sq. in.) than the standard-finish slabs containing the base mix. On the other hand, the average decrease in the strength of the control beams is approximately 150 lb. per sq. in.

Although there was quite an appreciable difference in the grading of the two sands (see Table I) the strengths of the sections containing the coarse sand are not greatly different from corresponding sections containing fine sand, although there is a tendency for somewhat lower strengths in the latter case. The same comment was made in connection with series A. Reference to Table III shows that in the case of gravel the concretes containing the fine sand contained one-tenth part less sand by volume than the corresponding coarse-sand concretes. In the case of crushed stone, the decrease in sand content was 0.2 part. These changes in sand content resulted in an increase in cement content not exceeding in any case 0.30 sack per cu. yd.

The average percentage of honeycomb, as shown in Fig. 15 (*a*), in all cases is less for the Johnson-finish slabs, the average being about 2 per cent as compared to 6 per cent for the standard finish. This difference in honeycomb is probably due primarily to differences in consistency. Referring back to Fig. 9, it will be noted that, under the conditions of these tests, the percentage of honeycomb in the standard-finish slabs was reduced to less than 3 per cent only by the use of a minimum slump of 2½ in. Referring again to Fig. 15 (*a*), it will be seen that the average slump of the standard-finish concrete was 2⅛ in. for proportion A, and 1⅞ in. for proportion B. The Johnson-finished concrete on the other hand showed an average slump of 3 in. in the case of proportion A, and 2⅞ in. in the case of proportion B. Knowing the effect of honeycombing on slab strength (see Figs. 6, 7, 8, 16, 17 and 18) the question arises: How much of the increased slab strength shown for the Johnson method was due to the fact that less honeycomb developed than in the standard-finish concrete and how much was due to the elimination of water? This particular point will be discussed in connection with the discussion of individual slab-strength results.

Figure 15 (*b*) shows data similar to Fig. 15 (*a*), except that in this case only the crushed-stone concrete is considered. The results are the average of sections containing the fine and coarse sand. The corresponding values for the gravel concrete are shown in the left-hand panel of Fig. 15 (*c*). In connection with the difference in the strengths shown by the crushed stone and the gravel concrete attention is called to the discussion of Fig. 4 under series A in which it was emphasized that this difference is due to the particular aggregates used in this work and does not express a general relation. A comparison indicates that the increase in strength shown for the Johnson mix is, in general, more marked in the case of the crushed-stone concrete. For instance, for proportion A the increase in core strength is about 700 lb. per sq. in. for crushed stone as compared with about 500 lb. per sq. in. for gravel. In the leaner mix the difference is more marked, being about 1600 lb. per sq. in. for the crushed-stone concrete as compared with about 550 lb. per sq. in. for the gravel concrete.

In flexure the differences are of the same order. For proportion A, the Johnson mix shows an increase for crushed stone of about 95 lb. per sq. in. as against approximately 55 lb. per sq. in. for the gravel concrete. For the leaner mix the difference is about 110 lb. per sq. in. for crushed-stone concrete as compared with only about 30 lb. per sq. in. for the gravel concrete. That the large increase shown for the crushed-stone concrete in the lean mix in favor of the Johnson method is due in part to variations in workability is evidenced by the relatively large amount of honeycomb (8 per cent) shown for standard-finish sections having this particular combination as compared with only 1½ per cent for the corresponding Johnson mix. Here again the question arises as to how much of the difference in slab strength is due to difference in honeycomb and how much to the elimination of water by the Johnson-finishing method. This question, however, cannot be raised in the case of the cores because special efforts were made to secure only sound, uniform cores free from honeycomb. It has already been shown that the crushing strengths of cores taken from the Johnson slabs averaged about 13 per cent higher in strength than cores from the corresponding standard-finish sections.

In the right-hand panel of Fig. 15 (*c*) have been plotted the results of tests on the concrete containing aggregates graded similarly to the Platte River gravel (see Table I). Two mixes were used, 1:4 and 1:4½ by volume. This aggregate contained practically no material over ½ in. in size and consequently the concrete was not honeycombed in the same sense as the concretes containing normally graded coarse aggregate. Reference to the figure will show the same trends as regards strength as were noted in the case of the gravel and crushed-stone concrete. The increase in crushing strength for the Johnson-finished concrete averages about 1400 lb. per sq. in. for the two mixes, with the 1:4½ Johnson-finished concrete about 1000 lb. per sq. in. higher than the 1:4 standard-finish sections. In flexure the same trend is noted. Here the average strength of Johnson-finish slabs is about 70 lb. per sq. in. higher than the corresponding standard-finish slabs; with the 1:4½ Johnson-finish concrete about 50 lb. per sq. in. higher than the 1:4 standard-finish. Inasmuch as the Platte River concrete is to all intents and purposes a mortar and not a concrete at all, honeycombing due to segregation of coarse aggregate is entirely missing. Therefore, the question which may be raised in connection with the standard coarse-aggregate concrete as to the effect of honeycombing on strength would not apply. Nevertheless a very substantial increase in slab strength is noted. This fact, coupled with the undoubted effect of the Johnson method of finishing on crushing strength, leads to the conclusion that, entirely aside from the effect of honeycomb on strength, the flexural strength of concrete is increased by this method of manipulation.

In Figs. 16, 17 and 18, the relation between consistency, honeycomb and slab strength for each test slab in series B is shown. It will be seen, first, that the groups of slabs representing the Johnson-finish sections show, in general, somewhat higher strengths and somewhat less honeycomb than the corresponding standard-finish slabs. The relation between honeycomb and consistency may also be traced. In general, the sections substantially free from honeycomb are those in which the slump is greater than 2½ in. On the other hand, the sections containing the drier mixes (2-in. slump or less) are, in general, the ones which contain the most honeycomb. The adverse effect of honeycomb on the strength of individual slabs is very marked in the case of the crushed-stone concrete (see Fig. 17).

It will be of interest to compare the flexural strength of the standard-finished and Johnson-finished sections on the basis of those slabs which show less than 3 per cent honeycomb (the open circles of Figs. 16 and 17). Such a comparison should eliminate to a large degree the influence of honeycomb. On this basis it is possible to make 15 direct comparisons between the two methods of finishing; sections 77 S and 78 J cannot be compared due to the fact that all four slabs in section 77 S show more than 3 per cent honeycomb.

Referring to Figs. 16 and 17 it may be noted that 12 out of the

15 pairs of sections which can be compared show higher strengths for the Johnson finish than for the standard finish. For instance, the average strength of the two slabs represented by the open circles in section 68 J, Fig. 16, is higher than the average strength of the three slabs represented by the open circles in section 67 S, and so on. This would indicate that a part at least of the higher average flexural strength previously noted for the Johnson mix is due to the elimination of excess water and not to honeycomb. This conclusion is substantiated by reference to Fig. 18, where all of the slabs are free from honeycomb. Four comparisons can be made in this case, all of which show higher strengths for the Johnson finish.

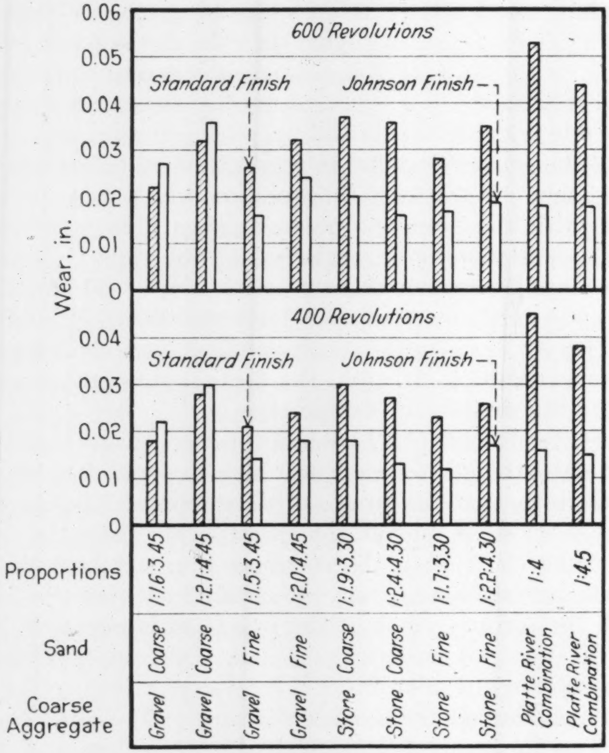

FIG. 19.—Effect of Johnson Method of Finishing on Surface Wear.

Analyzing Fig. 16 on the above basis, that is considering only those slabs which show less than 3 per cent honeycomb, it will be found that nine slabs in the four standard-mix gravel sections (Nos. 67, 71, 83 and 87) have an average flexural strength of about 720 lb. per sq. in. as compared with an average of approximately 680 lb. per sq. in. for twelve slabs in the four corresponding Johnson mix, Johnson-finish sections (Nos. 70, 74, 86 and 90). From Fig. 17 it will be found that eight slabs in sections Nos. 75, 79, 91 and 95 have an average flexural strength of approximately 915 lb. per sq. in. as against an average of about 890 lb. per sq. in. for fifteen slabs in sections Nos. 78, 82, 94 and 98. This is an average difference for the two types of aggregates of about 30 lb. per sq. in. or a little less than 4 per cent as compared with a drop, for the control beams representing these sections, of 150 lb. per sq. in. or roughly 20 per cent. A reduction in flexural strength of 150 lb. per sq. in. corresponds fairly closely to the reduction which would normally be expected from an increase in the water-cement ratio of 0.2, which is approximately the difference between the average water-cement ratio used in the standard mix, standard finish and the Johnson mix, Johnson finish (see Fig. 15 (*a*)). These figures show that entirely aside from the matter of honeycomb and its effect on strength, the Johnson-finish slabs in the leaner mix approached very nearly in strength the standard-finish slabs containing the richer mix.

*Surface Wear:*

The results of wear tests on the slabs in series B are shown graphically in Fig. 19. The data are plotted so as to show directly the depth of wear of the Johnson-finish slabs in comparison with the standard finish for each proportion and material combination. Each value is the average of two or three tests (one on each of two or three separate slabs). It will be observed that, in every case, with the exception of the gravel concrete with coarse sand, the Johnson-finish slabs show appreciably less wear than the standard finish. Just why, in this particular case, the reverse is true is not clear. However, the difference is not very marked, whereas, for several of the other combinations, particularly the crushed stone and coarse sand, and the Platte River concrete, there is a very distinct advantage in favor of the Johnson sections. The conclusions reached from these tests is that the Johnson method of delayed finishing certainly does not adversely affect the surface hardness of the pavement and very probably tends to increase it, due to the elimination of a portion of the water from the surface.

The results of these tests substantiate the observations which have been made on Johnson-finished pavements in service to the effect that the method of finishing employed in this process thoroughly incorporates the dry sand-cement mixture into the body of the concrete and does not cause scaling.

*Density and Absorption:*

Values for density and absorption of each material combination and for each method of finishing are shown in Fig. 20. Two values for density are shown, (1) the density of the plastic concrete calculated from the initial water content, and (2) the density of the hardened concrete calculated from the specific gravity of the cores.

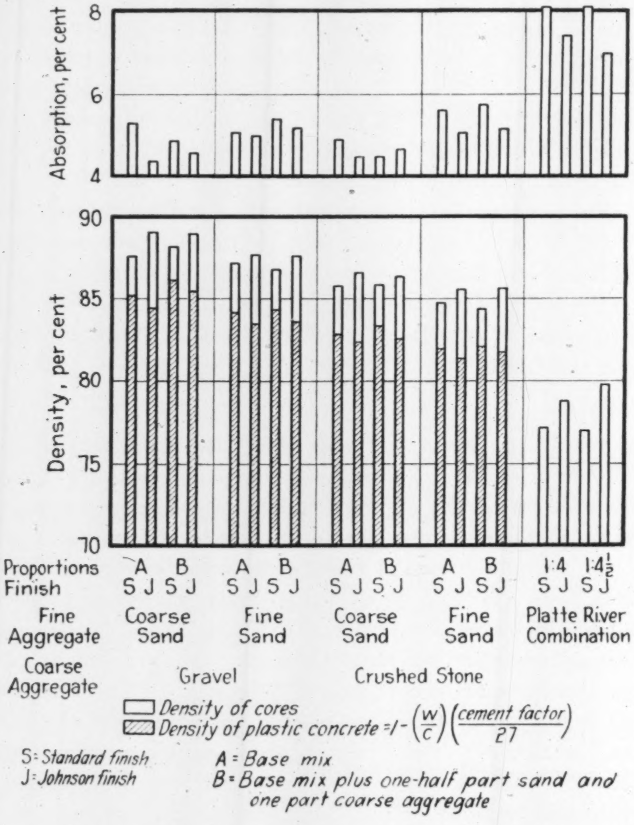

FIG. 20.—Density and Absorption Tests (Series B).

Inasmuch as the assumption that the plastic concrete is essentially free from air voids does not hold for the Platte River concrete, values for density calculated from water content have been omitted in this case. Comparing the density of each standard finished concrete with the corresponding concrete finished by the Johnson method we find that, whereas the density of the plastic concrete is greater in the case of the mixes which were finished by the standard method due to the fact that less water was used, the final density is greater in the case of the Johnson-finished concrete. This indicates that the Johnson method removed a considerably larger proportion of the total water before final consolidation took place than was accomplished by the standard method. This ties in very well with the increased crushing strength shown by the Johnson-finish cores.

This is shown graphically in Fig. 21. In this figure, the crushing strength of cylinders representing concrete used in the standard and Johnson-finished sections have been plotted against the corresponding initial water-cement ratios together with the results of core tests on the same concrete plotted against the void-cement ratios which were obtained from the values for density of the hardened concrete. Each point represents the average of 16 tests on 8 sections. It will be observed that, for both coarse aggregate types, all points, both cores and cylinders, lie substantially on a curve paralleling the conventional water-cement ratio - strength curve. It should be borne in mind that the core tests were made on the same concrete, in so far as proportions were concerned, as the corresponding cylinder tests. The core tests, however, were made on the concrete after final consolidation had taken place and a considerable amount of water removed. The cylinders, of course, were not subjected to the finishing operation and retained most of the water. These data appear to indicate quite definitely the reason for the increased strength of the cores as compared with the cylinders, as well as the increased strength of the

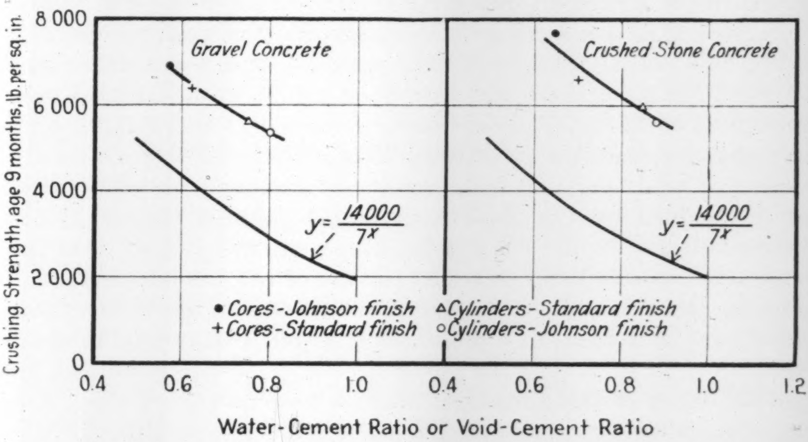

FIG. 21.—Relation Between Water-Cement Ratio, Void-Cement Ratio and Strength (Series B).

Johnson-finished cores as compared with the standard finish. It is all a matter of the final or net water-cement ratio which, in this case, seems to be measured quite accurately by the actual void-cement ratio of the cores as determined by test.

As indicated above, the Platte River concrete cannot be analyzed in this way due to the presence of air voids. Here the actual void-cement ratio of the plastic concrete is much higher than the water-cement ratio. Consequently the density bears no relation to relative water content.

On the assumption that the difference between the initial water-cement ratio and the final void-cement ratio is a measure of water loss during consolidation, it will be of interest to compare the average difference for each finishing method and for each type of coarse aggregate. Reference to Fig. 21 will show that this average difference in the case of the Johnson-finished gravel concrete was 0.23 (from 0.57 to 0.80) as against 0.13 for the standard-finished gravel concrete (0.62 to 0.75). The difference between 0.13 and 0.23, or 0.10, may therefore be said to represent the additional decrease in the volume of voids per unit bulk volume of cement (the void-cement ratio) obtained by the application of the Johnson method. The corresponding value for the crushed-stone concrete is 0.08. On this basis, it seems safe to assume that the use of the Johnson method of finishing is equivalent, in so far as improvement in strength and density are concerned, to a reduction in water-cement ratio of about 0.10.

The effect of this additional increase in density on the ultimate durability of the concrete has not been investigated, although it is planned to make a series of tests as a continuation of the present investigation. All available information on the subject indicates that, for a given mix, a very definite relationship exists between density and durability. It would seem therefore that any process which would result in an increase in density would be desirable from this standpoint alone, entirely aside from the effect on strength.

## CONCLUDING STATEMENT

The major facts developed by this investigation many be summarized as follows:

*Series A—Effect of Vibration:*

1. When finished by the standard method, the minimum slump required to insure pavement slabs substantially free from honeycomb was found to be approximately 2½ in.

2. When finished by vibration, the minimum slump required

to insure the same degree of uniformity was found to be approximately 1 in. (In the previous investigation it was found that standard-finished pavement slabs constructed of concrete having a 1-in. slump were less uniform in quality than similar slabs in which 2- to 3-in. slump concrete was used.)

3. The average flexural strength of all vibrated pavement slabs in which the concrete showed an average slump of 1 in. was somewhat higher than the average strength of the standard-finished concrete of the same proportions in which the average slump was 2 in. (In the previous investigation standard-finished slabs constructed of 1-in. slump concrete were generally lower in flexural strength than similar concrete having a 2-in. slump.)

4. The average increase noted under (3) was due to the marked increase in strength for the group of sections in which crushed stone was used as coarse aggregate. For the sections containing gravel as coarse aggregate, the 1-in. slump vibrated concrete showed a lower average flexural strength than the 2-in. slump standard-finished concrete. In the case of slag, the strengths for the two slumps were about the same.

5. The average flexural strength of all vibrated pavement slabs in which the concrete showed an average slump of $\frac{1}{2}$ in. was lower than the average strength of the standard-finished concrete of the same proportions in which the slump averaged 2 in.

6. The average decrease noted under (5) was due to the marked decrease in strength for the group of sections in which gravel was used as coarse aggregate. In the case of stone and slag, the average strength of the $\frac{1}{2}$-in. slump concrete, vibrated, was about the same as the average strength of the 2-in. slump standard-finish sections.

7. The average flexural strength of all vibrated pavement slabs constructed of concrete having the same proportions of cement, fine aggregate and water but containing one-fourth part more coarse aggregate by volume than the standard-finished concrete, was considerably higher than the standard-finished concrete. (In the previous investigation, concrete containing more coarse aggregate by volume than the base mix when finished by the standard method showed lower flexural strengths than the base mix, the decrease being roughly proportioned to the amount of coarse aggregate added.)

8. The increase noted under (7) was most marked in the case of the crushed-stone concrete and least in the case of the gravel concrete.

9. The average flexural strength of all vibrated concrete slabs containing one-half part more coarse aggregate than the standard-

finished concrete was somewhat higher than the standard-finished concrete. The sections containing crushed stone were considerably higher, the sections containing slag slightly higher and the sections containing gravel lower.

10. The average flexural strength of all vibrated concrete slabs containing three-fourths part more coarse aggregate than the standard-finished concrete was lower than the standard-finished concrete. The sections containing crushed stone and slag were about the same, whereas the sections containing gravel showed a decrease.

11. For concrete having the same water-cement ratio, finishing by vibration did not increase the crushing strength of cores drilled from the pavement slabs containing gravel and slag as coarse aggregate. In the case of crushed stone a considerable increase in crushing strength was noted for the vibrated concrete containing one-half and three-fourths parts additional coarse aggregate.

12. The process of finishing by vibration did not adversely affect the hardness of the surface.

*Series B—Effect of Delayed Finishing* (*Johnson Method*):

1. Pavement slabs finished by the Johnson method showed higher crushing and flexural strengths than standard-finished concrete of the same proportion but mixed with less water. This observation applies to both of the proportions studied.

2. Pavement slabs finished by the Johnson method developed substantially the same crushing and flexural strengths as standard-finished slabs containing one-half part less fine aggregate and one part less coarse aggregate. The Johnson-finished slabs, after correcting for the amount of cement in the "dry mix," contained approximately one sack of cement less per cubic yard of concrete than the standard-finished slabs.

3. Although, for each proportion, the water-cement ratio at the time of mixing was lower in the case of the standard-finished concrete, the final density of the concrete as revealed by tests on the cores was higher in the case of the Johnson-finished slabs.

4. Variations in crushing and flexural strength due to changes in proportions and methods of finishing were more marked in the crushed stone concrete than in the gravel concrete.

5. The process of finishing by the Johnson method did not adversely affect the hardness of the concrete.

6. The process of finishing by the Johnson method resulted in decreasing the voids-cement ratio approximately 0.1 more than the corresponding decrease shown for the standard-finish concrete.

## CONCLUSIONS

It is believed that the following conclusions are justified:

1. By the use of vibrating equipment of the same general type as employed in connection with these tests it should be possible to satisfactorily place and finish considerably drier concrete than is possible with methods now in common use.

2. For conditions comparable with these tests, it should be possible to satisfactorily place and finish concrete by vibration having a minimum slump of 1 in. as compared to a minimum slump of $2\frac{1}{2}$ in. for methods now in common use.

3. These tests indicate that, depending on the type of coarse aggregate used, concrete containing from one-fourth to three-fourths parts more coarse aggregate than the base mix, if vibrated, should show as great uniformity and as high flexural strength as the base mix finished by methods now in common use. Such a mix will contain approximately from 0.2 to 0.6 sack of cement per cu. yd. less than the base mix.

4. The indications of these tests are that the effect of vibration is more marked where angular coarse aggregates are used than where aggregates having rounded surfaces are used.

5. The use of the Johnson method of finishing as carried out in these tests will increase the density and strength of concrete pavement slabs by eliminating a larger quantity of excess water before final consolidation than is accomplished by the methods now in common use.

6. The application of the Johnson method of finishing to a mix containing approximately one-half part more fine and one part more coarse aggregate than the base mix will produce pavement slabs having substantially the same crushing and flexural strength as the base mix finished by methods now in common use. Such a mix will contain approximately one sack of cement per cubic yard of concrete less than the base mix.

7. In general, the indications of these tests are that the proper use of either the vibratory method of finishing or the Johnson method of finishing should result in an improvement in the quality of concrete pavements.

# DISCUSSION

**Mr. T. C. Powers.**[1]—Subsequent to the work at the U. S. Bureau of Public Roads, the vibrating machine on which Mr. Jackson reported was tested in Texas, Missouri, Illinois, and Michigan. It was my privilege to observe the tests in the last three states.

In general, we found very much the same results that Mr. Jackson has reported. From a general comparison of results I believe that in the later field tests, when a 20-ft. screed was used, the vibration was a little more effective. By appropriate changes in consistency, together with a lower proportion of sand, the water content of the concrete was reduced about 4 gal. per cu. yd. in most instances. That, on a constant water-cement ratio basis, corresponds to a reduction of almost a sack of cement per cubic yard.

In other respects the field tests confirm Mr. Jackson's findings.

**Mr. A. D. Conrow.**[2]—I should like to ask Mr. Jackson if he has made a study of the economics of the Johnson method of placing concrete in pavements. From the description given, the Johnson method seems somewhat complicated. This might mean that the cost of the extra manipulation required to obtain the improved concrete may be greater than would be needed to obtain it by other means, such as use of a somewhat greater quantity of cement.

**Mr. F. H. Jackson.**[3]—We did not make any attempt to study the relative cost of placing the "Johnson" type of pavement as compared with the standard method. We were concerned only with the relative quality of the product and made no attempt to measure either the time or cost of placing the slabs.

**Mr. F. C. Lang.**[4]—What is Mr. Jackson's opinion as to the desirability of having the subgrade absorb part of the excess water in the Johnson method? Would the use of tar paper be detrimental under any conditions?

**Mr. Jackson.**—It is probable that the character and moisture condition of the subgrade did influence the amount of moisture loss to a considerable extent. A well-compacted clay subgrade, such as we had at Arlington, probably draws a relatively small amount of water out of the concrete as compared with a sandy soil. In laying "Johnson" pavements the practice is to have the subgrade as dry as possible just prior to depositing the concrete in order to promote moisture loss. The usual practice of wetting down the subgrade just before placing the concrete is not followed.

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[2] Special Engineer, Ash Grove Lime and Portland Cement Co., Chanute, Kans.

[3] Senior Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[4] Engineer of Tests, Inspection and Research, Minnesota Highway Dept., St. Paul, Minn.

MR. P. D. MIESENHELDER.[1]—I should like to ask Mr. Jackson if any test was made that indicated the Johnson method drew the water throughout the entire depth of the slab, or if it was only from the upper portion? Also, did the finished surface of the slab form the compression side in the flexural test?

MR. JACKSON.—We made no attempt to measure the relative amount of moisture loss at various depths. However, the flexure tests were made on beams tested with the bottom in tension. If the increase in strength which was noted in the "Johnson" sections was due to moisture loss, it would appear that water was either lost through the subgrade or was actually drawn through the entire depth of slab, indicating that the loss was not confined to the surface only. The average moisture loss was not determined directly but was ascertained by comparing the density of the hardened concrete cores to the density of the plastic concrete when first placed.

MR. A. N. TALBOT.[2]—May I ask what relation the amount of cement put on the top bears to the total amount of cement used in the slab? Does the volume of water which would normally be taken by the added cement compare fairly well with the amount of water probably eliminated from the concrete below, and was a considerable amount of water drained off of the top? To what extent was the water-cement ratio of the slab decreased by the operation?

MR. JACKSON.—The average water-cement ratio of the concrete going into the sections finished by the "Johnson" method was approximately 0.84. This ratio was calculated on the basis of the actual amount of cement in the mix when laid and does not include the amount added through the finishing operation, which amounted to approximately one-fourth bag of cement per cubic yard of concrete. Taking this additional cement into consideration, the water-cement ratio would be reduced approximately to 0.80. However, as indicated in the discussion of Fig. 21, the "Johnson" method of finishing apparently reduced the water-cement ratio of the Johnson slabs approximately 0.10 more than the corresponding reduction in the standard finished slabs. This would indicate that considerably more water was eliminated by the finishing operation than could be accounted for by the addition of cement. This water was eliminated either by absorption in the subgrade, which in this case was probably very small, or by evaporation during the two or three hours required for the finishing operation.

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[2] Professor Emeritus, University of Illinois, Urbana, Ill.

# STRENGTH, WATER ABSORPTION AND WEATHER RESISTANCE OF BUILDING BRICKS PRODUCED IN THE UNITED STATES[1]

By J. W. McBurney[2] and C. E. Lovewell[2]

## Synopsis

This paper reports averages and distribution for flat compressive strength, modulus of rupture and water absorption by three methods for brick samples from 255 plants whose output represented 37 per cent of the 1929 brick production of the United States. The attempt is made to correlate these data with the manufacturers' classifications of the samples into "hard" and "salmon" as well as with the results of 51 cycles of freezing and thawing of approximately 480 specimens, mostly salmon.

## Introduction

This paper presents, in part, the results of a survey, made by the Common Brick Manufacturers' Association of America in cooperation with the U. S. Bureau of Standards, of certain properties of brick produced in the United States. The survey included securing from the manufacturers various items of information such as details of method of manufacture, percentage of the kiln output represented by each grade and the manufacturer's opinion as to the resistance of each grade to frost action as well as reports as to complaints about efflorescence and wet walls. The laboratory work included determinations of compressive strength, modulus of rupture, water absorption by several methods, size, weight, and tendency to effloresce. Rather complete descriptions of each individual brick were recorded, including exterior and interior color, presence or absence of nodules and inclusions, degree of lamination and nature of grain. The results on color have been previously reported.[3] It is intended to discuss size and weight in another paper.

The original plan was to subject each brick to an indefinite number of freezing-and-thawing cycles. Circumstances required the suspension of the work after completing 51 cycles on 480 specimens, classified for the most part as salmon brick.

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[1] Publication approved by the Director of the Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate of the Common Brick Manufacturers' Assn. at the U. S. Bureau of Standards, Washington, D. C.

[3] J. W. McBurney, "The Color Range of Common Brick," *Journal*, Clay Products Inst. of America, Vol. I, No. 2, June, 1932, pp. 31–40.

Table I.—Description of Brick Samples and Districts Sampled.

| District | Percentage of 1930 Sales Represented by Cities | Percentage of 1929 Production Represented by Districts | Number of Plants Sampled | Number of Samples | Number of Specimens | Raw Material[b] | Method of Forming[b] |
|---|---|---|---|---|---|---|---|
| Maine, New Hampshire and Vermont | .... | 62 | 12 | 45 | 261 | C | SM |
| Massachusetts, except Boston |  | 71 | 8 | 26 | 131 | C | SM |
| Boston, Mass | 85 |  | 5 | 16 | 94 | C | SM, SC[c] |
| Connecticut | .... | 77 | 11 | 45 | 236 | C | SM |
| Hudson Valley | 59 | 70 | 18 | 49 | 278 | C | SM |
| New York, except Hudson Valley |  |  | 3 | 6 | 45 | S, C | SM, SC |
| New Jersey | .... | 23 | 9 | 32 | 183 | C | SM, SC, EC |
| Eastern Pennsylvania, except Philadelphia |  | 56 | 13 | 43 | 218 | S, C, M | SM, SC |
| Philadelphia, Pa | 90 |  | 8 | 28 | 140 | C | SM, EC |
| Delaware | .... | .... | 2 | 10 | 50 | C | SM, EC |
| Maryland, except Baltimore |  | .... | 1 | 4 | 20 | C |  |
| Baltimore | 67 |  | 4 | 13 | 65 | C | SM, SC, EC |
| Pittsburgh, Pa | 70 | 55 | 8 | 23 | 138 | S | SC |
| West Virginia and Western Pennsylvania, except Pittsburgh |  |  | 3 | 11 | 59 | S, C | SC |
| Cleveland, Ohio | 80 | 79 | 3 | 10 | 50 | S, C | SM, SC |
| Northern Ohio, except Cleveland |  |  | 9 | 23 | 139 | S, C, FC | SM, SC, EC |
| Detroit | 75 | .... | 6 | 19 | 95 | C | SM |
| Wisconsin | .... | 21 | 6 | 21 | 94 | C | SM, SC |
| Chicago | 72 | 65 | 10 | 11 | 138 | C | EC |
| Illinois, except Chicago |  |  | 5 | 15 | 81 | S, C, M | SC, DP |
| St. Louis | 60 | 18 | 5 | 15 | 87 | S, C | SC, DP |
| Missouri, except St. Louis |  |  | 2 | 3 | 15 | S | SC |
| Kentucky | .... | 27 | 5 | 14 | 91 | C | SM, SC, DP |
| Southern Indiana | .... | .... | 3 | 3 | 18 | S, C, M | SM, SC |
| Nebraska | .... | 46 | 5 | 13 | 65 | S, C | SM, SC |
| Kansas | .... | .... | 1 | 8 | 40 | S | SC |
| Denver, Colorado | 60 | 57 | 3 | 8 | 50 | S, C | SC, DP |
| Colorado, except Denver |  |  | 5 | 15 | 83 | S | SC, DP |
| Utah and Wyoming | .... | .... | 2 | 12 | 60 | C, S, FC, M | SC, EC, DP |
| Washington and Oregon | .... | .... | 3 | 7 | 35 | C | SM, SC |
| Los Angeles, Calif | 90 | 78 | 13 | 13 | 65 | [d] | [e] |
| California, except Los Angeles |  |  | 19 | 19 | 95 | [f] | [g] |
| Texas | .... | 48 | 15 | 30 | 247 | S, C | SC, EC, DP |
| Mississippi, Louisiana and Arkansas | .... | 28 | 8 | 17 | 94 | S, C | SM, SC, EC, DP |
| Alabama, Tennessee and N. Carolina | .... | 16 | 12 | 29 | 164 | S, C, FC, M | SC, EC, DP |
| Richmond, Va | 95 | 12 | 5 | 14 | 68 | C | SC, SM |
| Virginia, except Richmond |  |  | 3 | 8 | 44 | S, C |  |
| Washington, D. C | 75 | .... | 2 | 6 | 50 | C | SM, SC, SDP |
| Total |  | 37.4[a] | 255 | 684 | 3886 |  |  |

[a] Percentage of 1929 production of the United States.

[b] C = clay. S = shale. FC = fire clay. M = mixture. SM = soft mud. SC = side cut stiff mud. EC = end cut stiff mud. DP = dry press. SDP = semi-dry press (containing more moisture than ordinary dry press).

[c] One plant only uses side-cut process.

[d] No information, but from water absorption the raw material is evidently clay.

[e] No information.

[f] Information on all 19 plants lacking. Four plants report clay or shale as raw material.

[g] Information on all 19 plants lacking. Four plants report soft mud or side-cut process of forming.

Except for some discussion of the relation of strength, water absorption and the ratio, 48 hr. cold to 5 hr. boiling water absorption, to frost resistance, there is no attempt, because of space limitations, to interpret the data given or to discuss their relation.

## Method of Sampling

Each manufacturer was requested to submit a sample consisting of not fewer than five brick for each commercial grade as classified and marketed by him. Where soft or salmon (underburned) brick were produced, a special sample representing that grade was requested, whether such brick were marketed or reburned. Information as to the percentage of the kiln product represented by each sample was in all cases secured from the manufacturer. If it were the manufacturer's practice to sell the entire output of the kiln without sorting or classifying, then a sample of 15 brick was requested. It was suggested that this "kiln run" sample should be selected so as to represent a vertical section of the kiln setting. The range of properties shown by the samples indicated that the majority of the manufacturers submitted the two extremes of their product. However, the survey provided no check on the accuracy of the manufacturer's estimates of the percentage of production represented by each sample. The question of geographic location of the plant having an influence on the manufacturer's classification of his product into "hard" (well burned) and "salmon" (underburned) brick will be discussed under the section, "Presentation of Data on Weather Resistance of Brick."

## Methods of Test

The methods for determining compressive strength, modulus of rupture and water absorption of the bricks were essentially those of this Society.[1] The procedure was to dry each specimen in an oven at 105 C. for at least 48 hr. and determine the modulus of rupture. One of the resulting half-bricks was trimmed if necessary and capped with plaster of Paris (calcined gypsum) and tested in compression flatwise. The other half-brick was totally immersed in water at room temperature for 5 hr., weighed, reimmersed for an additional 43 hr., reweighed and finally boiled for 5 hr., allowing the specimen and water to cool overnight. The method of making freezing-and-thawing tests is fully described in another paper.[2]

## Presentation and Discussion of Data

Table I furnishes the background for the succeeding tables. Obviously an average or distribution representing 90 per cent of the production of a district is more likely to be trustworthy than where but one or two plants are represented. The percentages of produc-

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[1] Standard Method of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31), 1931 Supplement to Book of A.S.T.M. Standards, p. 65.

[2] J. W. McBurney, "The Weathering of Structural Clay Products: A Review," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 745 (1931).

TABLE II.—WEIGHTED AVERAGES OF STRENGTH, WATER ABSORPTION, AND RATIO, OF 48 HR. COLD TO 5 HR. BOILING WATER ABSORPTION, FOR HARD AND SALMON BRICKS BY DISTRICTS AND CITIES.

| District or City | Type of Brick | Average Compressive Strength, lb. per sq. in. | Average Modulus of Rupture, lb. per sq. in. | Average Water Absorption, per cent | | | Average Ratio, 48 hr. Cold to 5 hr. Boiling Water Absorption |
|---|---|---|---|---|---|---|---|
| | | | | 5 hr. Cold | 48 hr. Cold | 5 hr. Boiling | |
| Maine, New Hampshire and Vermont | Hard | 10 330 | 1505 | 5.7 | 6.3 | 8.7 | 0.70 |
| | Salmon | 5 520 | 995 | 13.9 | 14.5 | 17.2 | 0.84 |
| Boston, Mass | Hard | 8 240 | 1360 | 7.7 | 8.2 | 10.6 | 0.74 |
| | Salmon | 3 710 | 645 | 16.4 | 17.1 | 19.6 | 0.88 |
| Massachusetts, except Boston | Hard | 5 940 | 1100 | 9.8 | 10.3 | 13.2 | 0.77 |
| | Salmon | 3 410 | 700 | 15.7 | 16.4 | 19.8 | 0.84 |
| Connecticut | Hard | 7 210 | 1155 | 12.9 | 13.5 | 16.2 | 0.82 |
| | Salmon | 4 670 | 770 | 18.6 | 19.1 | 21.8 | 0.87 |
| Hudson Valley | Hard | 4 740 | 745 | 14.0 | 14.6 | 17.7 | 0.80 |
| | Salmon | 2 580 | 425 | 19.5 | 20.0 | 24.3 | 0.83 |
| New York, except Hudson Valley | Hard | 4 770 | 1005 | 14.4 | 15.4 | 18.3 | 0.83 |
| New Jersey | Hard | 5 410 | 900 | 9.1 | 10.0 | 13.6 | 0.73 |
| | Salmon | 2 080 | 530 | 17.7 | 18.9 | 22.1 | 0.86 |
| Delaware | Hard | 8 040 | 1210 | 6.9 | 7.7 | 12.0 | 0.63 |
| | Salmon | 2 800 | 450 | 12.9 | 13.8 | 16.0 | 0.86 |
| Baltimore, Md | Hard | 5 990 | 1330 | 8.0 | 9.1 | 12.3 | 0.75 |
| | Salmon | 3 130 | 645 | 17.3 | 18.2 | 20.9 | 0.88 |
| Maryland, except Baltimore | Hard | 4 440 | 960 | 8.4 | 9.2 | 13.4 | 0.68 |
| | Salmon | 2 680 | 570 | 14.1 | 15.4 | 18.5 | 0.83 |
| Philadelphia, Pa | Hard | 6 580 | 855 | 8.9 | 9.8 | 14.5 | 0.67 |
| | Salmon | 2 520 | 355 | 16.7 | 17.9 | 22.9 | 0.79 |
| Eastern Pennsylvania, except Philadelphia | Hard | 8 080 | 1415 | 7.8 | 8.7 | 11.8 | 0.73 |
| | Salmon | 3 250 | 580 | 17.0 | 18.0 | 21.3 | 0.82 |
| Pittsburgh, Pa | Hard | 11 060 | 2055 | 4.8 | 5.5 | 7.7 | 0.69 |
| | Salmon | 4 880 | 955 | 11.4 | 12.1 | 14.1 | 0.85 |
| West Virginia and Western Pennsylvania, except Pittsburgh | Hard | 11 450 | 1845 | 4.9 | 5.6 | 8.0 | 0.69 |
| | Salmon | 4 810 | 940 | 11.7 | 12.5 | 14.6 | 0.85 |
| Cleveland, Ohio | Hard | 9 820 | 1185 | 8.2 | 8.7 | 11.1 | 0.78 |
| | Salmon | 7 780 | 1285 | 9.7 | 10.3 | 12.0 | 0.85 |
| Northern Ohio, except Cleveland | Hard | 12 110 | 1720 | 7.1 | 7.9 | 10.4 | 0.68 |
| | Salmon | 3 910 | 620 | 12.6 | 13.5 | 14.6 | 0.79 |
| Detroit, Mich | Hard | 4 330 | 900 | 15.6 | 16.5 | 19.5 | 0.81 |
| | Salmon | 2 540 | 555 | 18.9 | 19.4 | 20.8 | 0.93 |
| Wisconsin | Hard | 5 330 | 1110 | 15.6 | 16.6 | 20.3 | 0.81 |
| | Salmon | 2 320 | 470 | 24.4 | 25.2 | 28.0 | 0.90 |
| Chicago, Ill | Hard | 3 450 | 1265 | 13.2 | 15.2 | 19.0 | 0.79 |
| | Salmon | 2 400 | 420 | 20.0 | 22.4 | 26.5 | 0.85 |
| Illinois, except Chicago | Hard | 13 050 | 1580 | 5.2 | 6.2 | 8.0 | 0.75 |
| | Salmon | 6 330 | 780 | 14.3 | 15.2 | 17.2 | 0.88 |
| Kentucky | Hard | 7 780 | 1255 | 10.3 | 11.2 | 15.5 | 0.73 |
| | Salmon | 4 010 | 720 | 14.7 | 15.8 | 19.0 | 0.84 |
| Southern Indiana | Hard | 16 280 | 2360 | 1.8 | 2.3 | 5.4 | 0.37 |
| St. Louis, Mo | Hard | 11 040 | 1465 | 6.0 | 6.7 | 9.1 | 0.70 |
| | Salmon | 5 940 | 585 | 13.8 | 14.7 | 16.3 | 0.90 |
| Missouri, except St. Louis | Hard | 14 660 | 2020 | 2.9 | 3.8 | 5.7 | 0.61 |
| | Salmon | 6 430 | 1170 | 12.9 | 13.8 | 15.8 | 0.88 |
| Nebraska | Hard | 8 300 | 1025 | 8.7 | 9.7 | 13.3 | 0.73 |
| | Salmon | 2 480 | 460 | 14.0 | 15.3 | 20.6 | 0.76 |
| Kansas | Hard | 15 850 | 1915 | 3.2 | 3.8 | 5.1 | 0.59 |
| | Salmon | 4 500 | 600 | 15.2 | 16.0 | 17.6 | 0.91 |
| Denver, Colorado | Hard | 5 050 | 620 | 10.4 | 11.3 | 13.5 | 0.82 |
| | Salmon | 4 210 | 415 | 13.6 | 14.4 | 16.4 | 0.88 |
| Colorado, except Denver | Hard | 7 340 | 1145 | 10.3 | 11.1 | 13.3 | 0.83 |
| | Salmon | 4 900 | 740 | 15.5 | 16.0 | 17.8 | 0.90 |
| Utah | Hard | 7 490 | 1305 | 13.0 | 14.2 | 16.6 | 0.78 |
| Wyoming | Hard | 6 310 | 1410 | 7.2 | 8.1 | 12.1 | 0.68 |
| | Salmon | 5 680 | 300 | 13.4 | 13.9 | 16.3 | 0.85 |
| Washington and Oregon | Hard | 7 600 | 1245 | 9.5 | 10.3 | 14.1 | 0.70 |
| | Salmon | 3 240 | 580 | 15.9 | 17.0 | 20.1 | 0.85 |
| Los Angeles, Calif | Hard | 3 690 | 600 | 11.2 | 11.8 | 14.2 | 0.83 |
| California, except Los Angeles | Hard | 4 640 | 735 | 11.9 | 12.7 | 15.2 | 0.84 |
| Texas | Hard | 7 285 | 1020 | 11.1 | 12.0 | 14.3 | 0.82 |
| Mississippi, Louisiana and Arkansas | Hard | 6 320 | 911 | 11.4 | 12.3 | 16.5 | 0.79 |
| | Salmon | 2 580 | 485 | 10.1 | 11.1 | 16.8 | 0.67 |
| Alabama, Tennessee and North Carolina | Hard | 9 700 | 1525 | 7.0 | 7.9 | 10.6 | 0.72 |
| | Salmon | 4 510 | 750 | 17.6 | 18.9 | 21.0 | 0.89 |
| Richmond, Va | Hard | 9 350 | 1315 | 7.4 | 8.3 | 11.1 | 0.74 |
| | Salmon | 4 120 | 650 | 15.0 | 15.9 | 18.0 | 0.88 |
| Virginia, except Richmond | Hard | 9 470 | 1815 | 6.7 | 7.5 | 10.0 | 0.75 |
| | Salmon | 6 560 | 1440 | 11.2 | 11.9 | 13.8 | 0.87 |
| Washington, D. C | Hard | 5 700 | 875 | 8.3 | 9.4 | 12.9 | 0.72 |
| | Salmon | 3 500 | 385 | 14.3 | 15.4 | 16.7 | 0.91 |
| Weighted average of all samples, both hard and salmon | | 7 246 | 1154 | 10.12 | 10.92 | 13.99 | 76.4 |
| Weighted average of all Hard Samples | | 7 434 | 1183 | 9.77 | 10.56 | 13.66 | 75.8 |
| Weighted average of all Salmon Samples | | 4 094 | 678 | 15.94 | 16.77 | 19.41 | 85.1 |

tion represented for the several states and for the entire United States were furnished by the Bureau of the Census. The percentages of production represented by the samples from the several cities were provided by the Common Brick Manufacturers' Association. That portion of the United States situated between the Mississippi River and the Rocky Mountains as well as several sections of the South are poorly represented. Among the departures from accuracy of sampling by this survey can be listed the nonrepresentation of northern New Jersey and western Maryland shale bricks. The peculiarly characterized shale bricks from Minnesota are also not represented. However, the reasonableness of the survey would appear to be somewhat established: first, on account of the consistency and resemblance of samples from different plants in the same district and, second, by the very wide range in properties represented, considering the survey as a whole.

Table II deals with weighted averages. The weighting used in calculating these averages considers only the percentage of the kiln which each sample represents. The fact that different plants may have different sizes of kilns, different burning times, and different numbers of kilns, in other words, widely different capacities and rates of production, has been ignored. It was found impossible to get the complete and comparable data necessary to weight on a production basis.

The grand averages given at the end of Table II would be lowered for compressive strength, slightly changed for modulus of rupture and raised for water absorption in consideration of Chicago and the Hudson Valley being relatively large producers of brick. The question of production weighted averages may, however, be considered as more academic than practical. A shipment of brick is usually the product of one plant, not a weighted assortment from several plants.

The question of the interpretation of the terms "hard" and "salmon," as applied to brick grades in Table II and thereafter throughout the paper, warrants some consideration. This represents the manufacturer's grading into "satisfactory" and "unsatisfactory" from the standpoint of weathering resistance. It became evident early in the survey that certain parts of the United States which boast climate (not weather) produced no salmon bricks. This matter of climatic conditions influencing the manufacturer's grading of his product is considered under the section "Presentation of Data on Weather Resistance of Brick." It might be said in passing that the subject of manufacturers' grade names for brick merits a separate paper.

Another comment on Table II deals with the reproducibility of the data. Barring a change in the method of forming or a radical change in the nature of the raw material, the conclusion from repeated sampling of certain plants, together with a study of test data from other laboratories, is that kiln range samples are highly reproducible. In previous reports[1,2] on bricks, sampled and tested in 1926 and 1927, some 15 plants were represented which were again sampled in the present survey. In thirteen out of fifteen comparisons of samples, the agreement was as good as would be expected on resamples from the same shipment. Of the two remaining plants, one had substituted down draft for scove kilns between samplings. No information is available on the remaining plant.

TABLE III.—DISTRIBUTION OF PROPERTIES OF "HARD BRICKS" FROM ALL PARTS OF THE UNITED STATES COVERED BY SURVEY.

"Hard Bricks" are those so classified by the manufacturer.

| Compressive Strength, Flatwise | | Modulus of Rupture | | Water Absorption | | | | Ratio, 48 hr. Cold to 5 hr. Boiling Water Absorption | |
|---|---|---|---|---|---|---|---|---|---|
| Range, lb. per sq. in. | Percentage of Production Within Range | Range, lb. per sq. in. | Percentage of Production Within Range | Range, per cent | Percentage of Production Within Range | | | Range | Percentage of Production Within Range |
| | | | | | 5 hr. Cold | 48 hr. Cold | 5 hr. Boiling | | |
| 21 001 to 22 500 | 0.46 | 2101 to 3450 | 6.95 | 0 to 2.00 | 3.87 | 2.65 | 0.53 | 0.16 to 0.30 | 0.37 |
| 19 501 to 21 000 | 0.69 | 1951 to 2100 | 3.00 | 2.01 to 4.00 | 8.05 | 4.96 | 2.77 | 0.31 to 0.35 | 0.45 |
| 18 001 to 19 501 | 0.46 | 1801 to 1950 | 2.74 | 4.01 to 6.00 | 14.99 | 13.00 | 3.27 | 0.36 to 0.40 | 0.21 |
| 16 501 to 18 000 | 2.04 | 1651 to 1800 | 7.57 | 6.01 to 8.00 | 13.73 | 14.22 | 9.94 | 0.41 to 0.45 | 0.64 |
| 15 001 to 16 500 | 1.49 | 1501 to 1650 | 8.34 | 8.01 to 10.00 | 16.52 | 15.82 | 13.77 | 0.46 to 0.50 | 1.28 |
| 13 501 to 15 000 | 3.71 | 1351 to 1500 | 5.34 | 10.01 to 12.00 | 11.80 | 11.77 | 11.19 | 0.51 to 0.55 | 1.17 |
| 12 001 to 13 500 | 4.76 | 1201 to 1350 | 7.12 | 12.01 to 14.00 | 11.14 | 15.29 | 13.88 | 0.56 to 0.60 | 3.30 |
| 10 501 to 12 000 | 7.78 | 1051 to 1200 | 10.55 | 14.01 to 16.00 | 8.84 | 8.24 | 13.09 | 0.61 to 0.65 | 8.64 |
| 9 001 to 10 500 | 8.61 | 901 to 1050 | 10.44 | 16.01 to 18.00 | 5.01 | 5.52 | 11.02 | 0.66 to 0.70 | 12 37 |
| 7 501 to 9 000 | 11.92 | 751 to 900 | 13.60 | 18.01 to 20.00 | 2.42 | 4.77 | 9.14 | 0.71 to 0.75 | 16.80 |
| 6 001 to 7 500 | 15.47 | 601 to 750 | 11.74 | 20.01 to 22.00 | 2.22 | 2.31 | 5.72 | 0.76 to 0.80 | 19.41 |
| 4 501 to 6 000 | 16.81 | 451 to 600 | 7.52 | 22.01 to 24.00 | 1.31 | 0.64 | 2.14 | 0.81 to 0.85 | 15.65 |
| 3 001 to 4 500 | 17.97 | 301 to 450 | 4.35 | 24.01 to 26.00 | 0.00 | 0.68 | 1.95 | 0.86 to 0.90 | 13.89 |
| 1 501 to 3 000 | 7.46 | 151 to 300 | 0.37 | 26.01 to 28.00 | 0.11 | 0.00 | 0.75 | 0.91 to 0.95 | 5.32 |
| 0 to 1 500 | 0.36 | 0 to 150 | 0.37 | 28.01 to 34.00 | 0.00 | 0.11 | 0.75 | 0.96 to 1.00 | 0.51 |
| Total per cent . | 99.99 | | 100.00 | | 100.01 | 99.98 | 99.91 | | 100.01 |

It will be noted that figures for compressive strengths have been rounded off to the nearest 10 lb. per sq. in. and moduli of rupture to the nearest 5 lb. per sq. in.

Table II gives average values for properties of the bricks from several districts. Table III gives the dispersion of values of these same tests for all the hard brick averaged in Table II. Table III is chiefly useful in showing the wide range of strength and water absorption exhibited by the brick production of the United States. Table IV

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[1] J. W. McBurney, "The Compressive and Transverse Strength of Brick," U. S. Bureau of Standards *Journal of Research*, Vol. 2, April, 1929, p. 821.

[2] J. W. McBurney, "The Water Absorption and Penetrability of Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 711 (1929).

## TABLE IV.—DISTRIBUTION AS PER CENT OF TOTAL SAMPLES FROM EACH CITY.

The samples included only hard bricks as classified by the manufacturer.

| Range | Boston | New York | Philadelphia | Baltimore | Washington | Richmond | Pittsburgh | Cleveland | Detroit | Chicago | St. Louis | Denver | Los Angeles |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **COMPRESSIVE STRENGTH, HALF BRICK FLATWISE** | | | | | | | | | | | | | |
| LB. PER SQ. IN.: | | | | | | | | | | | | | |
| 16 501 to 18 000 | | | | | | | | | | | 9.8 | | |
| 15 001 to 16 500 | | | | | | | 9.4 | | | | | | |
| 13 501 to 15 000 | 17.5 | | | | | 15.2 | 19.8 | | | | | | |
| 12 001 to 13 500 | 2.1 | | | | | | 11.4 | 4.4 | | | 24.4 | | |
| 10 501 to 12 000 | 14.8 | | 1.6 | | | 15.2 | 9.4 | 63.2 | 2.1 | | 19.5 | | |
| 9 001 to 10 500 | 2.1 | | 13.2 | 19.7 | 35.9 | 14.8 | 29.9 | 1.9 | 10.5 | | 25.3 | | |
| 7 501 to 9 000 | 2.1 | | 33.5 | 21.3 | | 27.6 | 6.7 | | | | | 8.3 | 7.7 |
| 6 001 to 7 500 | 23.4 | 10.0 | 21.1 | 8.5 | 7.5 | 27.2 | 13.4 | 30.5 | | | 21.0 | 27.1 | |
| 4 501 to 6 000 | 19.0 | 55.5 | 30.6 | | 22.6 | | | | 12.8 | | | 29.2 | 15.3 |
| 3 001 to 4 500 | 19.0 | 27.8 | | 50.5 | | | | | 57.6 | 79.3 | | 35.4 | 38.5 |
| 1 501 to 3 000 | | 6.7 | | | 34.0 | | | | 17.0 | 20.7 | | | 38.5 |
| Total per cent | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Percentage 1930 sales represented by samples | 85.0 | 59.0 | 90.0 | 66.7 | 75.0 | 95.0 | 70.0 | 80.0 | 75.0 | 72.5 | 60.0 | 60.0 | 90.0 |
| **MODULUS OF RUPTURE** | | | | | | | | | | | | | |
| LB. PER SQ. IN.: | | | | | | | | | | | | | |
| 2 551 to 3 300 | | | | | | | 6.7 | | | | | | |
| 2 401 to 2 550 | 7.0 | | | | | | 4.0 | | 2.1 | | | | |
| 2 251 to 2 400 | | | | 1.1 | | | 21.8 | | 2.1 | | 9.8 | | |
| 2 101 to 2 250 | | | | | | | 9.4 | | | | | | |
| 1 951 to 2 100 | 2.1 | | | | | | 15.2 | 2.2 | 2.1 | | | | |
| 1 801 to 1 950 | | | | 21.3 | | | 14.8 | 0.04 | | 10.6 | 19.5 | | |
| 1 651 to 1 800 | 12.7 | | | 18.6 | | 15.2 | 4.7 | 3.6 | 6.3 | 19.2 | | | |
| 1 501 to 1 650 | 14.8 | | | | | 16.0 | 21.4 | 25.4 | 6.8 | | 8.8 | | |
| 1 351 to 1 500 | 14.8 | | | 6.6 | 35.9 | | 2.0 | 1.8 | | | 18.8 | 8.3 | |
| 1 201 to 1 350 | | 6.7 | 3.9 | 8.5 | | 29.2 | | | 2.1 | 19.2 | 24.4 | | 7.7 |
| 1 051 to 1 200 | 9.1 | 8.0 | 17.1 | 18.6 | | 14.0 | | 30.5 | | 9.6 | 11.8 | | |
| 901 to 1 050 | 23.2 | 4.9 | 22.6 | | 7.5 | 25.6 | | | 3.9 | 20.7 | | 16.7 | |
| 751 to 900 | 16.3 | 18.9 | 24.2 | 9.3 | | | | 36.5 | 13.1 | 10.6 | | 10.4 | 7.7 |
| 601 to 750 | | 43.3 | 23.9 | 16.0 | 22.6 | | | | 13.1 | 10.1 | 6.9 | | 23.0 |
| 451 to 600 | | 12.6 | 3.1 | | 34.0 | | | | 48.4 | | | | 30.8 |
| 301 to 450 | | 5.6 | 5.2 | | | | | | | | | 64.6 | 30.8 |
| Total per cent | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| **WATER ABSORPTION, 5 HR. IN COLD WATER** | | | | | | | | | | | | | |
| PER CENT: | | | | | | | | | | | | | |
| 0 to 2.00 | 9.1 | | | 1.1 | | | 6.8 | 0.04 | 6.4 | | 9.8 | | |
| 2.01 to 4.00 | 12.7 | 0.3 | | | | | 27.9 | 2.9 | 6.3 | | 19.5 | | |
| 4.01 to 6.00 | 27.4 | 0.3 | 4.7 | 18.6 | 35.9 | 20.6 | 30.5 | 29.1 | 6.8 | | 33.3 | 8.3 | |
| 6.01 to 8.00 | 4.2 | 1.2 | 27.2 | 21.2 | 7.5 | 40.9 | 34.8 | 37.4 | 2.1 | | 9.2 | 16.7 | |
| 8.01 to 10.00 | 14.8 | 7.4 | 32.7 | 47.9 | 22.6 | 38.5 | | | | 19.7 | 9.6 | 10.4 | 30.7 |
| 10.01 to 12.00 | 21.1 | 15.1 | 27.6 | 11.2 | 34.0 | | | | | 10.6 | 18.6 | 29.2 | 38.5 |
| 12.01 to 14.00 | 10.7 | 16.0 | 7.8 | | | | | 30.6 | 3.9 | 20.2 | | 35.4 | 23.1 |
| 14.01 to 16.00 | | 38.0 | | | | | | | 9.2 | 28.7 | | | |
| 16.01 to 18.00 | | 16.8 | | | | | | | 3.9 | 20.8 | | | |
| 18.01 to 20.00 | | 4.9 | | | | | | | 35.3 | | | | |
| 20.01 to 22.00 | | | | | | | | | 16.9 | | | | 7.7 |
| 22.01 to 24.00 | | | | | | | | | 9.2 | | | | |
| Total per cent | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| **WATER ABSORPTION, 48 HR. IN COLD WATER** | | | | | | | | | | | | | |
| PER CENT: | | | | | | | | | | | | | |
| 0 to 2.00 | 2.1 | | | | | | 6.7 | 0.3 | 6.4 | | 9.8 | | |
| 2.01 to 4.00 | 9.1 | | | 1.1 | | | 11.4 | 0.7 | | | 19.5 | | |
| 4.01 to 6.00 | 38.0 | 0.3 | 1.6 | | | 2.3 | 34.3 | 5.8 | 6.3 | | 28.3 | 8.3 | |
| 6.01 to 8.00 | | 0.5 | 13.2 | 39.9 | 43.4 | 59.1 | 36.9 | 62.9 | | | 4.9 | | |
| 8.01 to 10.00 | 19.0 | 3.2 | 49.9 | 6.6 | | 26.2 | 10.7 | | 8.9 | 9.6 | 18.8 | 16.7 | 25.0 |
| 10.01 to 12.00 | | 12.7 | 11.4 | 50.5 | 22.6 | 12.4 | | | | 10.1 | | 39.6 | 8.3 |
| 12.01 to 14.00 | 21.1 | 22.0 | 19.2 | 1.9 | 34.0 | | | 30.6 | | 20.2 | 18.7 | | 58.4 |
| 14.01 to 16.00 | 10.7 | 29.3 | 4.7 | | | | | | 13.1 | 10.7 | | 35.4 | 8.3 |
| 16.01 to 18.00 | | 27.1 | | | | | | | | 28.7 | | | |
| 18.01 to 20.00 | | 4.9 | | | | | | | 35.3 | 20.7 | | | |
| 20.01 to 22.00 | | | | | | | | | 20.8 | | | | |
| 22.01 to 24.00 | | | | | | | | | 9.2 | | | | |
| Total per cent | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

TABLE IV.—(*Continued.*)

| Range | Boston | New York | Philadelphia | Baltimore | Washington | Richmond | Pittsburgh | Cleveland | Detroit | Chicago | St. Louis | Denver | Los Angeles |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| WATER ABSORPTION, 5 HR. IN BOILING WATER | | | | | | | | | | | | | |
| PER CENT: | | | | | | | | | | | | | |
| 0 to 2.00 | 2.1 | .... | .... | .... | .... | .... | .... | 0.04 | 2.1 | .... | .... | .... | .... |
| 2.01 to 4.00 | .... | .... | .... | 1.1 | .... | .... | 6.7 | .... | 4.3 | .... | 9.8 | .... | .... |
| 4.01 to 6.00 | 9.1 | .... | .... | .... | .... | .... | 3.3 | .... | 6.3 | .... | .... | .... | .... |
| 6.01 to 8.00 | 10.6 | 0.3 | .... | .... | .... | 1.6 | 42.4 | 32.0 | .... | .... | 28.3 | 8.3 | .... |
| 8.01 to 10.00 | 27.4 | .... | .... | 39.8 | 43.4 | 33.8 | 36.9 | 35.7 | .... | .... | 24.4 | 16.7 | .... |
| 10.01 to 12.00 | 19.0 | 0.5 | 16.4 | .... | .... | 26.1 | 10.7 | 1.8 | .... | .... | 18.8 | 39.6 | 8.3 |
| 12.01 to 14.00 | .... | 3.2 | 41.3 | 31.9 | 22.6 | 26.1 | .... | .... | 8.9 | 9.6 | 6.9 | .... | 33.3 |
| 14.01 to 16.00 | 21.1 | 13.2 | 13.2 | 1.9 | .... | 12.4 | .... | .... | .... | .... | 11.8 | 35.4 | 41.7 |
| 16.01 to 18.00 | 10.7 | 24.5 | 16.1 | 25.3 | .... | .... | .... | 30.5 | .... | 30.3 | .... | .... | 16.7 |
| 18.01 to 20.00 | .... | 35.1 | 13.0 | .... | 34.0 | .... | .... | .... | 3.9 | 10.6 | .... | .... | .... |
| 20.01 to 22.00 | .... | 23.2 | .... | .... | .... | .... | .... | .... | 30.0 | 39.4 | .... | .... | .... |
| 22.01 to 24.00 | .... | .... | .... | .... | .... | .... | .... | .... | 18.4 | .... | .... | .... | .... |
| 24.01 to 26.00 | .... | .... | .... | .... | .... | .... | .... | .... | 16.9 | 10.1 | .... | .... | .... |
| 26.01 to 28.00 | .... | .... | .... | .... | .... | .... | .... | .... | 9.2 | .... | .... | .... | .... |
| Total per cent | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

gives the strength and water absorption ranges for the hard brick of thirteen cities of the United States. Other distribution data are given in Tables V and VI, discussed in the following section.

## PRESENTATION OF DATA ON WEATHER RESISTANCE OF BRICK

The problem here considered is the possibility of satisfactorily predicting the weathering resistance of brick from strength and water absorption determinations, with or without the use of other measures. The data at hand include: first, the manufacturer's own opinion of the weatherability of various grades of bricks, produced and used in a wide variety of climates; second, laboratory determinations of strength and water absorption upon these grades; and third, a series of freezing and thawing tests, for the most part upon brick classified as salmon.

The weathering resistance under consideration is practically synonymous with resistance to frost action. Disintegration through frost action depends upon the occurrence of repeated freezings and thawings of more or less wetted brick. Obviously, therefore, the climate must be such that temperatures below freezing occur during part of the year and there must be sufficient precipitation to have the bricks wetted part of the time. The northeastern quarter of the United States has the combination of freezing temperature and moisture. The limits, north and south, were set by the January mean 32 F. isotherm. This line enters the United States near New York City, proceeds south parallel to the coast until near Baltimore and then extends west, running a little north of the Ohio River. A

TABLE V.—DISTRIBUTION OF PROPERTIES OF BRICKS FROM NORTHEASTERN QUARTER OF UNITED STATES.

"Hard" and "Salmon" bricks are those so classified by the manufacturer.

| Compressive Strength, Flatwise | | | Modulus of Rupture | | | Water Absorption | | | | | | | Ratio, 48 hr. Cold to 5 hr. Boiling Water Absorption | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Range, lb. per sq. in. | Percentage of Production Within Range | | Range, lb. per sq. in. | Percentage of Production Within Range | | Range, per cent | Percentage of Production Within Range | | | | | | Range | Percentage of Production Within Range | |
| | | | | | | | 5 hr. Cold | | 48 hr. Cold | | 5 hr. Boiling | | | | |
| | Hard | Salmon | | Hard | Salmon | | Hard | Salmon | Hard | Salmon | Hard | Salmon | | Hard | Salmon |
| 21 001 to 22 500... | 0.72 | .... | 2101 to 3450..... | 8.86 | .... | 0 to 2.00.... | 5.55 | .... | 3.52 | .... | 0.83 | .... | 0.16 to 0.30..... | 0.49 | .... |
| 19 501 to 21 001... | 1.01 | .... | 1951 to 2100..... | 3.70 | .... | 2.01 to 4.00... | 9.82 | .... | 6.44 | .... | 3.87 | .... | 0.31 to 0.35..... | 0.72 | .... |
| 18 001 to 19 501... | 0.74 | .... | 1801 to 1950..... | 3.15 | .... | 4.01 to 6.00... | 14.91 | .... | 14.32 | .... | 4.57 | .... | 0.36 to 0.40..... | 0.16 | .... |
| 16 501 to 18 001... | 2.79 | .... | 1651 to 1800..... | 8.87 | 0.11 | 6.01 to 8.00... | 10.96 | .... | 12.41 | .... | 10.77 | .... | 0.41 to 0.45..... | 0.59 | .... |
| 15 001 to 15 501... | 2.33 | .... | 1501 to 1650..... | 8.06 | 0.14 | 8.01 to 10.00... | 12.92 | 0.37 | 13.26 | 0.16 | 13.35 | 0.29 | 0.46 to 0.50..... | 1.98 | .... |
| 13 501 to 15 000... | 2.95 | .... | 1351 to 1500..... | 4.14 | 0.11 | 10.01 to 12.00... | 8.61 | 0.61 | 8.34 | 0.72 | 8.13 | 0.25 | 0.51 to 0.55..... | 1.55 | .... |
| 12 001 to 13 500... | 5.32 | 0.03 | 1201 to 1350..... | 6.96 | 0.03 | 12.01 to 14.00... | 7.99 | 1.42 | 9.99 | 1.27 | 9.20 | 1.03 | 0.56 to 0.60..... | 3.73 | .... |
| 10 501 to 12 000... | 8.04 | .... | 1051 to 1200..... | 9.34 | 0.58 | 14.01 to 16.00... | 10.19 | 1.35 | 8.60 | 1.29 | 8.40 | 1.13 | 0.61 to 0.65..... | 9.78 | 0.01 |
| 9 001 to 10 500... | 8.78 | 0.14 | 901 to 1050..... | 9.01 | 1.26 | 16.01 to 18.00... | 4.15 | 1.40 | 6.17 | 0.90 | 10.62 | 1.48 | 0.66 to 0.70..... | 10.75 | 0.003 |
| 7 501 to 9 000... | 7.14 | 0.32 | 751 to 900..... | 11.66 | 0.69 | 18.01 to 20.00... | 3.25 | 1.92 | 4.61 | 2.22 | 7.98 | 0.71 | 0.71 to 0.75..... | 14.63 | 0.34 |
| 6 001 to 7 500... | 13.33 | 1.09 | 601 to 750..... | 10.32 | 1.32 | 20.01 to 22.00... | 2.93 | 0.63 | 3.62 | 1.04 | 7.45 | 1.42 | 0.76 to 0.80..... | 19.19 | 1.91 |
| 4 501 to 6 000... | 16.95 | 1.24 | 451 to 600..... | 6.02 | 1.76 | 22.01 to 24.00... | 0.54 | 0.19 | 0.55 | 0.27 | 3.37 | 1.25 | 0.81 to 0.85..... | 17.36 | 1.97 |
| 3 001 to 4 500... | 17.17 | 2.22 | 301 to 450..... | 1.94 | 1.05 | 24.01 to 26.00... | 0.00 | 0.03 | 0.00 | 0.03 | 2.49 | 0.29 | 0.86 to 0.90..... | 8.92 | 2.62 |
| 1 501 to 3 000... | 4.70 | 2.73 | 151 to 300..... | 0.00 | 0.94 | 26.01 to 28.00... | 0.17 | 0.00 | 0.00 | 0.00 | 0.77 | 0.05 | 0.91 to 0.95..... | 1.93 | 1.04 |
| 0 to 1 500... | 0.00 | 0.24 | 0 to 150..... | 0.00 | 0.00 | 28.01 to 34.00... | 0.00 | 0.11[a] | 0.17[a] | 0.12[a] | 0.17 | 0.11 | 0.96 to 1.00..... | 0.23 | 0.11 |
| | 91.97 | 8.01 | | 92.03 | 7.99 | | 91.99 | 8.03 | 92.00 | 8.02 | 91.97 | 8.01 | | 92.01 | 8.003 |
| Total ............ | 99.98 | | | 100.02 | | | 100.02 | | 100.02 | | 99.98 | | | 100.013 | |

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[a] In range 28.01 to 30.00.

## TABLE VI.—COMPARISON OF DISTRIBUTION OF PROPERTIES FOR BRICKS MADE FROM CLAY AND FROM SHALE.

Samples are separated as hard and salmon according to the manufacturer's classification. Clay brick samples are from New England. The shale brick samples are from Pennsylvania, West Virginia, Illinois, Missouri and Kansas.

| Compressive Strength, Flatwise | | | | | Modulus of Rupture | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Range, lb. per sq. in. | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | | Range, lb. per sq. in. | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | |
| | Hard | Salmon | Hard | Salmon | | Hard | Salmon | Hard | Salmon |
| 21 001 to 22 500 | .... | .... | 0.6 | .... | 2101 to 3300 | 5.5 | .... | 24.3 | .... |
| 19 501 to 21 000 | .... | .... | 3.4 | .... | 1951 to 2100 | 1.3 | .... | 11.3 | .... |
| 18 001 to 19 500 | .... | .... | 3.5 | .... | 1801 to 1950 | 1.5 | .... | 6.7 | .... |
| 16 501 to 18 000 | 0.6 | .... | 8.5 | .... | 1651 to 1800 | 7.6 | 0.5 | 21.6 | .... |
| 15 001 to 16 500 | 1.3 | .... | 4.6 | .... | 1501 to 1650 | 12.0 | 0.6 | 16.8 | .... |
| 13 501 to 15 000 | 2.4 | .... | 6.6 | .... | 1351 to 1500 | 8.3 | .... | 4.7 | .... |
| 12 001 to 13 500 | 10.3 | .... | 11.8 | 0.2 | 1201 to 1350 | 4.2 | .... | 4.0 | 0.2 |
| 10 501 to 12 000 | 4.1 | .... | 25.5 | .... | 1051 to 1200 | 16.9 | 0.9 | 3.2 | 1.9 |
| 9 001 to 10 500 | 11.6 | .... | 15.8 | .... | 901 to 1050 | 15.5 | 4.7 | 2.6 | 1.3 |
| 7 501 to 9 000 | 10.2 | 0.9 | 5.4 | .... | 751 to 900 | 6.6 | 1.6 | 0.3 | 0.4 |
| 6 001 to 7 500 | 18.3 | 3.1 | 9.8 | 1.7 | 601 to 750 | 2.2 | 2.3 | .... | 0.5 |
| 4 501 to 6 000 | 19.2 | 4.3 | .... | 1.2 | 451 to 600 | 2.7 | 4.0 | .... | 0.2 |
| 3 001 to 4 500 | 6.2 | 3.8 | .... | 1.4 | 301 to 450 | .... | 1.1 | .... | .... |
| 1 501 to 3 000 | 0.1 | 3.6 | .... | .... | 151 to 300 | .... | .... | .... | .... |
| 0 to 1 500 | .... | .... | .... | .... | 0 to 150 | .... | .... | .... | .... |
| | 84.3 | 15.7 | 95 5 | 4.5 | | 84.3 | 15.7 | 95.5 | 4.5 |
| Total | 100.0 | | 100.0 | | | 100.0 | | 100.0 | |

| 5 hr. Cold Water Absorption | | | | | 48 hr. Cold Water Absorption | | | |
|---|---|---|---|---|---|---|---|---|
| Range, per cent | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | |
| | Hard | Salmon | Hard | Salmon | Hard | Salmon | Hard | Salmon |
| 0 to 2.00 | 4.2 | .... | 13.7 | .... | 1.8 | .... | 12.1 | .... |
| 2.01 to 4.00 | 8.4 | .... | 20.8 | .... | 6.5 | .... | 13.2 | .... |
| 4.01 to 6.00 | 13.7 | .... | 35.2 | .... | 17.8 | .... | 31.4 | .... |
| 6.01 to 8.00 | 14.7 | .... | 17.8 | .... | 10.1 | .... | 24.8 | .... |
| 8.01 to 10.00 | 13.2 | .... | 5.5 | 0.6 | 16.4 | .... | 11.5 | 0.2 |
| 10.01 to 12.00 | 7.2 | 1.7 | 2.2 | 1.0 | 6.2 | 1.1 | .... | 1.4 |
| 12.01 to 14.00 | 11.1 | 3.1 | 0.3 | 2.2 | 8.2 | 3.3 | 2.5 | 1.6 |
| 14.01 to 16.00 | 7.8 | 2.2 | .... | 0.7 | 9.6 | 2.1 | .... | 1.2 |
| 16.01 to 18.00 | 1.8 | 3.8 | .... | .... | 5.0 | 1.7 | .... | .... |
| 18.01 to 20.00 | 1.8 | 3.4 | .... | .... | 1.3 | 6.0 | .... | 0.1 |
| 20.01 to 22.00 | 0.4 | 0.6 | .... | .... | 1.4 | 0.6 | .... | .... |
| 22.01 to 24.00 | .... | 0.9 | .... | .... | .... | 0.9 | .... | .... |
| 24.01 to 26.00 | .... | .... | .... | .... | .... | .... | .... | .... |
| 26.01 to 28.00 | .... | .... | .... | .... | .... | .... | .... | .... |
| 28.01 to 30.00 | .... | .... | .... | .... | .... | .... | .... | .... |
| | 84.3 | 15.7 | 95.5 | 4.5 | 84.3 | 15.7 | 95.5 | 4.5 |
| Total | 100.0 | | 100.0 | | 100.0 | | 100.0 | |

| 5 hr. Boiling Water Absorption | | | | | Ratio, 48 hr. Cold to 5 hr. Boiling Water Absorption | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Range, per cent | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | | Range | Distribution, per cent of Total Clay | | Distribution, per cent of Total Shale | |
| | Hard | Salmon | Hard | Salmon | | Hard | Salmon | Hard | Salmon |
| 0 to 2.00 | 0.9 | .... | 2.4 | .... | 0.26 to 0.30 | .... | .... | .... | .... |
| 2.01 to 4.00 | 1.4 | .... | 12.3 | .... | 0.31 to 0.35 | .... | .... | 4.0 | .... |
| 4.01 to 6.00 | 5.0 | .... | 6.1 | .... | 0.36 to 0.40 | .... | .... | 0.2 | .... |
| 6.01 to 8.00 | 11.5 | .... | 28.6 | .... | 0.41 to 0.45 | 0.6 | .... | .... | .... |
| 8.01 to 10.00 | 12.4 | .... | 31.5 | .... | 0.46 to 0.50 | 0.5 | .... | 4.0 | .... |
| 10.01 to 12.00 | 18.5 | .... | 9.7 | 0.1 | 0.51 to 0.55 | 1.2 | .... | 4.2 | .... |
| 12.01 to 14.00 | 4.2 | .... | 2.7 | 1.4 | 0.56 to 0.60 | 1.2 | .... | 8.3 | .... |
| 14.01 to 16.00 | 11.8 | 3.0 | 2.2 | 1.6 | 0.61 to 0.65 | 10.1 | .... | 7.9 | .... |
| 16.01 to 18.00 | 9.0 | 2.8 | .... | 1.4 | 0.66 to 0.70 | 12.6 | .... | 14.5 | .... |
| 18.01 to 20.00 | 4.0 | 2.7 | .... | .... | 0.71 to 0.75 | 13.4 | .... | 22.3 | .... |
| 20.01 to 22.00 | 4.1 | 1.6 | .... | .... | 0.76 to 0.80 | 14.1 | 3.2 | 12.7 | .... |
| 22.01 to 24.00 | 1.0 | 4.1 | .... | .... | 0.81 to 0.85 | 15.9 | 4.4 | 16.8 | 1.2 |
| 24.01 to 26.00 | 0.5 | 1.5 | .... | .... | 0.86 to 0.90 | 11.7 | 5.3 | 0.6 | 3.1 |
| 26.01 to 28.00 | .... | .... | .... | .... | 0.91 to 0.95 | 3.0 | 2.8 | .... | 0.2 |
| 28.01 to 30.00 | .... | .... | .... | .... | 0.96 to 1.00 | .... | .... | .... | .... |
| | 84.3 | 15.7 | 95.5 | 4.5 | | 84.3 | 15.7 | 95.5 | 4.5 |
| Total | 100.0 | | 100.0 | | | 100.0 | | 100.0 | |

north and south line running through the center of Kansas was taken as separating the wet East from the semiarid West. Table V presents distribution for a combination of districts on or north of the January mean 32 F. isotherm and east of central Kansas.

Table II, with the averages for hard and salmon bricks by districts, makes it evident that salmon brick are always weaker, in most cases have a higher water absorption, and, with the exception of the few samples from the Mississippi-Louisiana-Arkansas district, have higher absorption ratios (48 hr. cold to 5 hr. boiling water absorption) than the corresponding hard samples. It should be emphasized that this paper proposes no absolute definition of a salmon brick. Acceptance of manufacturers' classifications presupposes a definition something on the order of "A salmon brick is one which will not reasonably withstand the climatic conditions of the district where it is used." Failure to understand that manufacturers made their classifications on the basis of the way their bricks behaved in their own locality was responsible for considerable confusion in the early stages of the survey. The fact that a particular sample of brick has a 5-hr. boiling water absorption of 24 per cent, a compressive strength less than 3000 lb. per sq. in., a pale color, *and is completely disrupted by three cycles of freezing and thawing* does not classify it as a salmon in certain southern and western districts by the definition above quoted. This, then, is the reason why Table V presents only samples from the northeastern quarter of the United States.

The relatively few freezing-and-thawing tests available tend to modify the manufacturer's classification in two particulars: In the first case, where the manufacturer has classified as "hard," bricks which are distinguished by low compressive strength, high water absorption and high absorption ratio, the freezing-and-thawing cycles tend to cause considerable damage. In the second case freezing-and-thawing tests cause little damage to bricks classified by the manufacturers as "salmon" if the bricks have relatively high strength, low water absorption and low absorption ratio. In other words, when manufacturers' classifications fail to agree with strength and absorption values, freezing-and-thawing tests check the absorption and strength results rather than the manufacturer's classification. Using actual freezing-and-thawing tests would be more preferable than acceptance of manufacturer's classification, but there are too few freezing-and-thawing test data available.

The possible conclusions from Table V will be considered along with those from Tables VI to VIII.

Table VI compares the properties of hard and salmon bricks from the standpoint of effect of raw material, clay and shale.

Tables VII and VIII present results of 51 cycles of freezing and thawing on approximately 480 individual specimens. The method of test is described in another paper.[1] Breakage or loss in weight of more than three per cent counted as "unsatisfactory" under the test. These 480 specimens do not represent a cross-section or fair sample of the survey. With the exception of 14 bricks, all the specimens were selected because they were of doubtful weather resistance. A number of the high absorption, high absorption ratio, low strength bricks from southern and western districts are included. About half the salmon grades of Table V are in these tests.

Table VII.—Effect of 51 Cycles of Freezing and Thawing on Individual Bricks Classified According to Flat Compressive Strength and Ratio of 48 hr. Cold to 5 hr. Boiling Water Absorption.

S = satisfactory.
U = unsatisfactory (loss of more than 3 per cent in weight).
Figures are number of bricks in each classification.

| Range, Compressive Strength, Flatwise, lb. per sq. in. | 0 to 0.40[a] | | 0.40 to 0.60[a] | | 0.60 to 0.70[a] | | 0.70 to 0.75[a] | | 0.75 to 0.80[a] | | 0.80 to 0.85[a] | | 0.85 to 0.90[a] | | 0.90 to 0.95[a] | | 0.95 to 1.00[a] | | Total | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U |
| 0 to 1 500 | ... | ... | 0 | 1[b] | 4 | 3[b] | 1 | 0 | 1 | 6 | 3 | 12 | 0 | 5 | 0 | 1 | 0 | 2 | 9 | 30 |
| 1 501 to 2 500 | ... | ... | 1 | 0 | 6 | 2[b] | 1 | 0 | 9 | 7 | 10 | 13 | 6 | 15 | 2 | 7 | 0 | 2 | 35 | 46 |
| 2 501 to 3 500 | ... | ... | 1 | 0 | 2 | 0 | 1 | 0 | 3 | 0 | 11 | 9 | 18 | 39 | 4 | 14 | 1 | 3 | 41 | 65 |
| 3 501 to 4 500 | ... | ... | 1 | 0 | ... | ... | 3 | 1[b] | 12 | 0 | 7 | 7 | 13 | 20 | 9 | 24 | 1 | 0 | 46 | 52 |
| 4 501 to 6 000 | ... | ... | ... | ... | 1 | 0 | 3 | 0 | 8 | 0 | 21 | 8 | 17 | 15 | 3 | 11 | ... | ... | 53 | 34 |
| 6 001 to 8 000 | ... | ... | ... | ... | 1 | 0 | 1 | 0 | ... | ... | 13 | 0 | 14 | 4 | 3 | 1 | ... | ... | 32 | 5 |
| 8 001 to 12 000 | ... | ... | 2 | 0 | 2 | 0 | 2 | 0 | 1 | 0 | 2 | 0 | 5 | 1 | 1 | 1 | ... | ... | 15 | 2 |
| 12 001 to 15 000 | ... | ... | 2 | 0 | 3 | 0 | 2 | 0 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 7 | 0 |
| 15 000 + | 2 | 0 | 5 | 0 | 2 | 0 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 9 | 0 |
| Total | 2 | 0 | 12 | 1 | 21 | 5 | 14 | 1 | 34 | 13 | 67 | 49 | 73 | 99 | 22 | 59 | 2 | 7 | 247 | 234 |

[a] Range, ratio 48 hr. cold to 5 hr. boiling water absorption.
[b] From Hudson Valley.

## Discussion of Weather Resistance Data

Considering the data of Table V, the problem is to select the measure which will reject the least percentage of satisfactory grades and accept the least percentage of unsatisfactory grades. If the numerical value for a particular property be selected so that one per cent of the bricks classified as salmon pass, then the percentage of the bricks classified as hard which are rejected by this figure serves as a measure of the separation afforded. The values respectively which separate one per cent of the bricks under the salmon classification are a compressive strength of between 6000 and 7500 lb. per sq. in., a modulus of rupture of about 1050 lb. per sq. in., close to 12 per cent for 5 hr. cold and 48 hr. cold water absorption and between 12 and 14 per cent absorption by 5 hr. boiling. However, the percentages of rejection of hard bricks afforded by these measures range from 30 to nearly 50 per cent. Obviously the 5-hr. cold water absorption provides the best separation, but a rejection of approximately 30 per cent of the hard bricks renders this out of the question.

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[1] J. W. McBurney, "The Weathering of Structural Clay Products: A Review," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 745 (1931).

Considering in like manner the data of Table VI, the following conclusions are apparent:

1. Hard and salmon shale bricks are all quite sharply separated by any one of all six measures.

TABLE VIII.—EFFECT OF 51 CYCLES OF FREEZING AND THAWING ON INDIVIDUAL BRICKS CLASSIFIED ACCORDING TO 5 HR. BOILING WATER ABSORPTION AND RATIO OF 48 HR. COLD TO 5 HR. BOILING WATER ABSORPTION.

S = satisfactory.
U = unsatisfactory (loss of more than 3 per cent in weight).
Figures are numbers of bricks in each classification.

| Range, 5 hr. Boiling Water Absorption, per cent | 0 to 0.40[a] | | 0.40 to 0.60[a] | | 0.60 to 0.70[a] | | 0.70 to 0.75[a] | | 0.75 to 0.80[a] | | 0.80 to 0.85[a] | | 0.85 to 0.90[a] | | 0.90 to 0.95[a] | | 0.95 to 1.00[a] | | Total | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U | S | U |
| 0 to 5 | 2 | 0 | 7 | 0 | 1 | 0 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | 10 | 0 |
| 5 to 8 | ... | ... | 2 | 0 | 5 | 0 | 2 | 0 | 1 | 0 | 1 | 0 | ... | ... | ... | ... | ... | ... | 11 | 0 |
| 8 to 10 | ... | ... | ... | ... | 1 | 0 | 1 | 0 | ... | ... | 1 | 0 | ... | ... | ... | ... | ... | ... | 3 | 0 |
| 10 to 12 | ... | ... | ... | ... | 1 | 0 | 2 | 0 | ... | ... | 1 | 0 | 2 | 0 | ... | ... | ... | ... | 6 | 0 |
| 12 to 15 | ... | ... | ... | ... | ... | ... | 4 | 0 | 5 | 0 | 17 | 2 | 19 | 10 | 4 | 3 | ... | ... | 49 | 15 |
| 15 to 18 | ... | ... | 1 | 0 | 6 | 0 | 1 | 0 | 4 | 0 | 10 | 6 | 25 | 18 | 4 | 15 | 0 | 1 | 51 | 40 |
| 18 to 21 | ... | ... | 1 | 0 | 2 | 0 | 1 | 0 | 6 | 2 | 11 | 14 | 6 | 17 | 10 | 14 | 2 | 1 | 39 | 48 |
| 21 to 24 | ... | ... | ... | ... | 3 | 1[b] | 2 | 0 | 11 | 3 | 14 | 10 | 12 | 35 | 2 | 15 | 0 | 4 | 44 | 68 |
| 24 + | ... | ... | 1 | 1[b] | 2 | 4[b] | 1 | 1[b] | 7 | 10 | 12 | 18 | 9 | 20 | 2 | 12 | 0 | 1 | 34 | 67 |
| Total | 2 | 0 | 12 | 1 | 21 | 5 | 14 | 1 | 34 | 15 | 67 | 50 | 73 | 100 | 22 | 59 | 2 | 7 | 247 | 238 |

[a] Ratio, 48 hr. cold to 5 hr. boiling water absorption.
[b] From Hudson Valley.

2. Not less than 7500 lb. per sq. in. for compressive strength, not less than 1200 lb. per sq. in. for modulus of rupture, not more than 10 per cent water absorption measured as either 5 hr. cold or 48 hr. cold, not more than 12 per cent water absorption measured as 5 hr. boiling water absorption, and a figure between 0.81 and 0.85 for the ratio 48 hr. cold to 5 hr. boiling water, effects in each case a separation whereby less than one per cent of shale salmon bricks pass and less than 10 per cent of shale hard bricks are rejected.

3. The hard and salmon clay bricks overlap in all their properties.

4. Of the several measures considered, the absorption ratio, 48 hr. cold to 5 hr. boiling water, comes the nearest to applying equally to both shale and clay bricks.

With this last conclusion in mind, Tables VII and VIII were prepared to show the effect of two measures combined as criteria of weather resistance.

From the data of Table VII, it is concluded that ability to withstand 51 cycles of freezing and thawing is ensured by either of the following two combinations: A compressive strength of not less than 2500 lb. per sq. in. with an absorption ratio of not more than 0.80; or, a compressive strength of not less than 6000 lb. per sq. in. with an absorption ratio of not more than 0.85. From Table VIII, correspondingly, the figures are: not more than 18 per cent water absorption by 5 hr. boiling when the absorption ratio is not more than 0.80; or, not more than 12 per cent water absorption by 5 hr. boiling with a ratio of not more than 0.85.

## Summary and Conclusions

From the data here presented the following conclusions are considered justified:

1. The weighted averages for all samples, representing specimens from approximately 37 per cent of the brick production of the United States are: flat compressive strength 7246 lb. per sq. in.; modulus of rupture 1154 lb. per sq. in.; water absorption, 5 hr. cold 10.12 per cent, 48 hr. cold 10.92 per cent, 5 hr. boiling water 13.99 per cent, and ratio 48 hr. cold to 5 hr. boiling water 0.764.

2. The weighted averages for all samples classified by the manufacturers as hard are: flat compressive strength 7434 lb. per sq. in.; modulus of rupture 1183 lb. per sq. in.; water absorption, 5 hr. cold 9.77 per cent, 48 hr. cold 10.56 per cent, 5 hr. boiling water 13.66 per cent, and ratio 48 hr. cold to 5 hr. boiling water 0.758.

3. The weighted averages for all samples classified by the manufacturers as salmon are: flat compressive strength 4094 lb. per sq. in.; modulus of rupture 678 lb. per sq. in.; water absorption, 5 hr. cold 15.94 per cent, 48 hr. cold 16.77 per cent, 5 hr. boiling water 19.41 per cent, and ratio 48 hr. cold to 5 hr. boiling water 0.851.

4. For the United States as a whole, the range in compressive strength is from less than 1500 lb. per sq. in. to over 21,000 lb. per sq. in. and the range in 5 hr. boiling water absorption is from less than 2.0 per cent to 34.0 per cent.

5. Shale brick samples have a narrower range for all properties than is shown by clay brick samples.

6. No single measure of strength or water absorption will sharply separate salmon from hard bricks where both clay and shale samples are considered together.

7. Shale bricks can be quite sharply separated into hard and salmon (considering the climate of the northeastern section of the United States) by requiring either a compressive strength of not less than 7500 lb. per sq. in. or a modulus of rupture of not less than 1200 lb. per sq. in., or water absorption of not more than 10 per cent measured either by 5 hr. cold or 48 hr. cold immersion, or a water absorption of not more than 12 per cent after 5 hr. boiling.

8. The absorption ratio 48 hr. cold to 5 hr. boiling water is least affected by difference in raw material.

9. The combination, not less than 2500 lb. per sq. in. compressive strength with an absorption ratio of not more than 0.80, or a compressive strength of not less than 6000 lb. per sq. in. with an absorption ratio of not more than 0.85, insures the withstanding of 51 cycles of freezing and thawing.

10. Likewise the combination, not more than 18 per cent water absorption by 5 hr. boiling water with an absorption ratio of not more than 0.80, or a water absorption of not more than 12 per cent by 5 hr. boiling with an absorption ratio not exceeding 0.85, insures the withstanding of 51 cycles of freezing and thawing.

*Acknowledgments:*

Messrs. W. W. Harrison, Gifford Earnest, D. A. Parsons, E. E. W. Bowen and Samuel E. Wade assisted in the laboratory work and in making the calculations at various times. Special acknowledgment is made to Norman W. Kelch of the California Clay Products Institute for permission to use unpublished data on California plants. Joseph Ulmer of the Bureau of Foreign and Domestic Commerce supplied figures on percentage of production. The survey would have been impossible without the help of the staff of the Common Brick Manufacturers' Association. Space does not permit the listing of the more than 200 manufacturers who submitted samples.

# TESTS ON BRICK MASONRY BEAMS

By M. O. Withey[1]

## Synopsis

Data are given on the shear and bending strengths of twenty-five 8 by 12-in. reinforced brick beams tested under third-point loading over an 8-ft. span. Three widely different varieties of brick, and several variations in percentage of longitudinal steel between 0.5 and 2.3 per cent, as well as different percentages of stirrup reinforcement were used. The tests indicate that a high degree of flexural strength and shear strength can be developed in reinforced brick beams provided due attention is paid to mortar bond, coursing, amount, and arrangement of reinforcement, and filling of joints. Formulas for reinforced concrete design with appropriate constants can be used to calculate stresses and deflections of reinforced brick beams.

## Introduction

Although records of the use of reinforced brick masonry date back to the building of a tunnel under the Thames by Brunel in 1825, comparatively little is reported concerning this type of construction until 1923. At that time the Indian Government published the results of extensive tests made by Brebner. Since then considerable testing of beams and slabs has been undertaken in this country, mostly sponsored by the Common Brick Manufacturers Association and applications of this type of construction have been made in India, Japan, and the United States.[2]

It was the aim in making the program for the tests herein reported to include a rather wide range in the character of three varieties of brick, and in the percentages of longitudinal and stirrup reinforcements used in these tests. This procedure was adopted in order that some notion might be secured with regard to the flexural limitations of this type of masonry.

The work was done as a thesis under the author's direction by three civil engineering seniors, Messrs. L. E. Angoli, L. L. Krasin and B. F. Ludowise in the spring of 1932. These students deserve much credit for their careful work in testing and in supervising the work of the mason who fabricated the beams. Services of the mason and materials were provided through the cooperation of the Common

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[2] For bibliography, see *Report No. 5*, Committee on Reinforced Brick Masonry, Am. Ceramic Soc. (1932).

Brick Manufacturers Association; Con Geminer and Sons of Waupaca, Wis., and the Streator Brick Co. of Streator, Ill., provided brick; and the Concrete Engineering Co. of Chicago, Ill., donated the steel. The tests were made in the Materials Testing Laboratory of the University of Wisconsin.

## MATERIALS

*Steel:*

Excepting the $\frac{1}{4}$-in. rods used for stirrups, all reinforcement consisted of deformed bars of the type indicated in Table IV and with yield points shown in Table VII. The variability in yield point was caused by the use of some old stock in certain of the beams.

TABLE I.—STRENGTHS OF MORTAR FROM MASON'S BATCHES.

**Proportions were: 1 hydrated lime : 3 cement : 12 sand with $2\frac{1}{2}$ to 3 parts of water, by weight. Each result represents 5 or 6 tests.**

| Beams | Average Tensile Strength, lb. per sq. in. | | | | Compressive Strength | |
|---|---|---|---|---|---|---|
| | 3 days | 7 days | 14 days | 28 days | Age, days | Strength of 2 by 4-in. Cylinders, lb. per sq. in. |
| C7, C8 | 252 | 282 | .... | 354 | .... | .... |
| C5, C6 | 245 | 336 | 356 | .... | 13 | 2848 |
| C1 to C4 | 254 | 322 | .... | 297 | 7 | 2870 |
| W1, W3, W5, W7 | .... | 321 | 353 | 386 | 15 | 3036 |
| W2, W4, W6, W8, S1, S2 | 259 | 296 | 346 | 367 | 14 | 2742 |
| S3 to S8, C9 | 263 | 321 | 345 | 367 | 14 | 2670 |
| Average | 255 | 313 | 350 | 354 | .... | 2833 |

The yield point of the $\frac{1}{4}$-in. round rods used in stirrups was 58,900 lb. per sq. in. Percentages of steel in beams are based on actual areas of rods.

*Sand:*

Janesville sand of which 3 per cent, 76 per cent, and 99 per cent were retained on No. 14, No. 48 and No. 100 sieves, respectively, was used in the mortar for these tests. The fineness modulus of the sand was 2.0

*Cement and Lime:*

To expedite the curing period, high-early-strength portland cement was used in all tests. The tensile strength of 1:3 standard sand mortar briquets made and tested in standard manner averaged 303, 359, 431, and 482 lb. per sq. in. at ages of 1, 3, 7 and 28 days, respectively. Limate, an hydrated dolomitic lime, obtained from the Western Lime and Cement Co. was included to render the mortar more plastic and workable.

*Mortar:*

The quantities of materials for each batch of mortar were carefully weighed. The usual batch consisted of 20 lb. of hydrated lime, 60 lb. of cement, 240 lb. of Janesville sand, and 50 to 60 lb. of water. Sand and cement were first dry mixed and the water then added. Mixing was done by a skilled workman with a hoe in a metal-lined box. Results of tension and compression tests of samples of mortar taken from the mason's batches are given in Table I.

TABLE II.—STRENGTH TESTS ON BRICK AND BRICK PIERS.

In the cross bending tests the span was 7 in. Piers were 8 by 8 by 25 in. in size. Plaster of Paris bedments were used in all crushing tests.

| Kind of Brick | Position in Which Tested | Average Strengths Based on 5 Tests, lb. per sq. in. | | Averages of 3 or 5 Pier Tests, lb. per sq. in. | |
|---|---|---|---|---|---|
| | | Compression | Cross Bending (Modulus of Rupture) | Compression Strength | Modulus of Elasticity |
| Chicago | Flatwise | 2 586 | 971 | | |
| | Edgewise | 2 390 | .... | | |
| | Endwise | 5 550 | .... | 2690 | 2 400 000 |
| Waupaca | Flatwise | 12 500 | 2250 | | |
| | Edgewise | 5 860 | .... | | |
| | Endwise | 5 900 | .... | 2870 | 3 500 000 |
| Streator | Flatwise | 9 195 | 1110 | | |
| | Edgewise | 4 034 | .... | | |
| | Endwise | 3 970 | .... | 2090 | 2 100 000 |

TABLE III.—STRENGTH OF MORTAR JOINTS IN DOUBLE SHEAR TESTS.

Each average represents 5 tests.

| Kind of Brick | Condition | Age at Test, days | Moisture in Bricks, per cent | Shearing Strength, lb. per sq. in. |
|---|---|---|---|---|
| Chicago | Sprinkled 5 min. | 13 | 6.7 | 19[a] |
| | Sprinkled 20 min. | 13 | 8.4 | 69 |
| | Soaked overnight. | 18 | 12.1 | 66 |
| | Sprinkled 5 min. | 18 | 6.7 | 61 |
| Waupaca | As received | 7 | .... | 105 |
| | Sprinkled 10 min. | 7 | 2.5 | 136 |
| Streator | As received | 14 | .... | 106 |
| | Sprinkled 10 min. | 12 | 3.4 | 138 |

[a] First batch of specimens made.

*Brick:*

Chicago common, an end-cut clay brick, Waupaca face brick, a side-cut brick pierced by three $\frac{7}{8}$-in. holes normal to the wide faces, and Streator side-cut common brick pierced by sixteen $\frac{1}{2}$ by 1-in. cells normal to the wide faces were supplied for these tests. The average dimensions of the brick were: Chicago, 8.05 by 3.63 by 2.28 in.; Waupaca, 7.68 by 3.50 by 2.18 in.; Streator, 8.03 by 3.68

by 2.20 in. The average weights of single brick were: Chicago, 4.18 lb.; Waupaca, 4.23 lb.; Streator, 3.49 lb.

Absorption tests were made on 5 brick of each kind. After drying to constant weight in an electric oven at 220 F. (105 C.), the brick were immersed in water at 70 F. (21 C.) and weighed at various intervals of time. The average absorptions after 5 min., 30 min., and 48 hr., respectively, were: Chicago 7.7, 10.9, 15.2 per cent; Waupaca 2.2, 3.6, 5.0 per cent; Streator 2.9, 4.5, 6.1 per cent. Results of crushing and cross bending tests are given in Table II.

TABLE IV.—SCHEDULE OF BEAMS FABRICATED.

Age of beams $C_1$, $C_2$, $C_7$ and $C_8$ was 1 week; all others tested at 2 weeks. Nominal dimensions, 8 by 12 in. by 10 ft. Span 8 ft. Loads at third points of span. Values of breadth and effective depth are given in Table VIII.

| Kind of Brick | Beam | Condition of Brick | Moisture in Brick, per cent | Reinforcement in Beams | | |
|---|---|---|---|---|---|---|
| | | | | Longitudinal | Per cent | Shear |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* |
| Chicago | C1 | Sprinkled 10 min. | 7.2 | Four $\frac{3}{8}$-in. round | 0.57 | One $\frac{1}{4}$-in. Z at 6 in. |
| | C2 | Sprinkled 5 min. | 6.7 | Four $\frac{3}{8}$-in. round | 0.56 | One $\frac{1}{4}$-in. Z at 6 in. |
| | C4 | Sprinkled 5 min. | 6.7 | Four $\frac{3}{8}$-in. round | 0.61 | None |
| | C5 | Sprinkled 5 min. | 6.7 | Four $\frac{1}{2}$-in. round | 0.98 | Two $\frac{1}{4}$-in. Z at 3 in. |
| | C6 | Soaked overnight | 12.1 | Four $\frac{1}{2}$-in. round | 0.93 | Two $\frac{1}{4}$-in. Z at 3 in. |
| | C9 | Sprinkled 10 min. | 7.2 | Four $\frac{1}{2}$-in. square | 1.23 | One $\frac{3}{8}$-in. Z at 3 in. |
| | C3 | Sprinkled 20 min. | 8.4 | Four $\frac{3}{8}$-in. round | 0.62 | None |
| | C7 | Sprinkled 5 min. | 6.7 | Four $\frac{1}{2}$-in. square | 1.33 | One $\frac{3}{8}$-in. U at 8 in. |
| | C8 | Sprinkled 5 min. | 6.7 | Four $\frac{1}{2}$-in. square | 1.24 | One $\frac{3}{8}$-in. Z at 3 in. |
| Waupaca | W3 | As received | .... | Four $\frac{3}{8}$-in. round | 0.62 | One $\frac{1}{4}$-in. Z at 6 in. |
| | W4 | As received | .... | Four $\frac{1}{2}$-in. round | 1.00 | Two $\frac{1}{4}$-in. Z at 6 in. |
| | W5 | Sprinkled 10 min. | 2.5 | Four $\frac{1}{2}$-in. round | 1.04 | Two $\frac{1}{4}$-in. Z at 4 in. |
| | W6 | As received | .... | Four $\frac{1}{2}$-in. round | 1.02 | Two $\frac{1}{4}$-in. Z at 4 in. |
| | W7 | Sprinkled 10 min. | 2.5 | Four $\frac{1}{2}$-in. square | 1.41 | One $\frac{3}{8}$-in. Z at 3 in. |
| | W8 | As received | .... | Four $\frac{5}{8}$-in. square | 2.31 | One $\frac{3}{8}$-in. Z at 3 in. |
| | W1 | As received | .... | Four $\frac{3}{8}$-in. round | 0.66 | None |
| | W2 | Sprinkled 10 min. | 2.5 | Four $\frac{3}{8}$-in. round | 0.70 | None |
| Streator | S1 | As received | .... | Four $\frac{3}{8}$-in. round | 0.66 | None |
| | S2 | Sprinkled 10 min. | 3.4 | Four $\frac{3}{8}$-in. round | 0.66 | None |
| | S3 | As received | .... | Four $\frac{1}{2}$-in. round | 1.15 | None |
| | S4 | As received | .... | Four $\frac{1}{2}$-in. square | 1.29 | One $\frac{3}{8}$-in. Z at 3 in. |
| | S5 | As received | .... | Four $\frac{1}{2}$-in. round | 1.06 | Two $\frac{1}{4}$-in. Z at 4 in. |
| | S6 | As received | .... | Four $\frac{1}{2}$-in. round | 1.03 | Two $\frac{1}{4}$-in. Z at 6 in. |
| | S8 | As received | .... | Four $\frac{1}{2}$-in. square | 1.30 | Two $\frac{1}{4}$-in. Z at 4 in. |
| | S7 | As received | .... | Four $\frac{5}{8}$-in. square | 2.22 | One $\frac{3}{8}$-in. Z at 3 in. |

## AUXILIARY TESTS ON BRICK MASONRY

The compressive strength and the modulus of elasticity of the brick masonry were determined from tests of 8 by 8 by 25-in. piers in which the brick were laid on end in a vertical position and no joints were broken. These specimens were covered with wet burlap overlaid with a damp canvas until tested. In testing, deformations were read over 15-in. gage lengths on opposite sides of the specimens by wire-wound dials reading to 0.0001 in. The average strengths and the average values of the secant modulus of elasticity computed at one third the ultimate strength are also recorded in Table II.

The bond strengths of the mortar to the bricks were ascertained from double shear tests. These tests were made by bonding three brick with their wide faces in contact. Five of these shear test specimens were made with every batch of beams and cured in the manner indicated for the compression prisms. In testing, the outer bricks rested on end on knife edges and the middle brick was axially loaded on its top end through a 2-in. spherical seat. The average results for each set of shear specimens is given in Table III. In the tests of the specimens made of Chicago and those made of Waupaca brick, strengths of some of the individual specimens varied 50 to 60 per cent from the given averages.

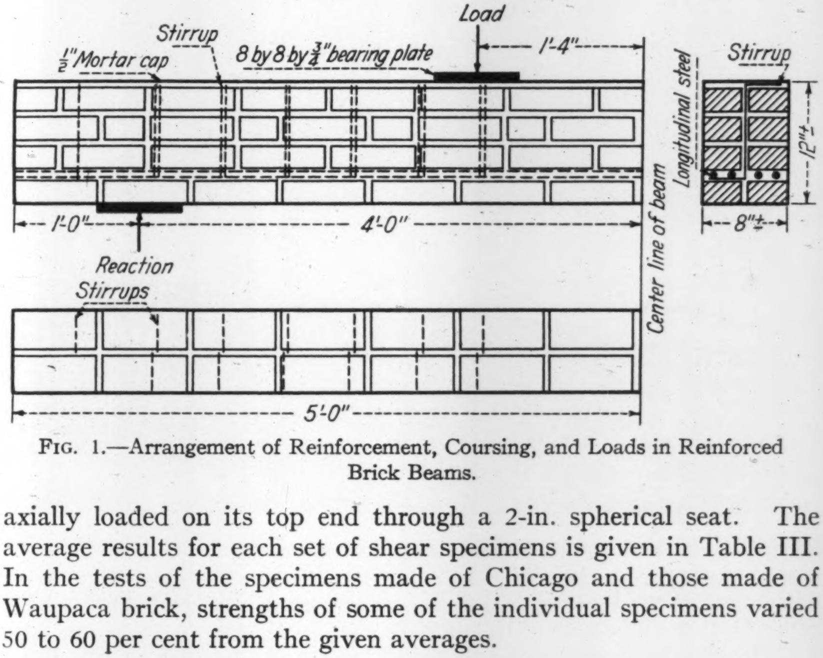


FIG. 1.—Arrangement of Reinforcement, Coursing, and Loads in Reinforced Brick Beams.

## MAKING OF BEAMS

Table IV gives the schedule of the beams which were fabricated. Prior to laying, the brick were given the treatment indicated in column 3 of the table. Beams were laid by an experienced bricklayer on oiled 10-in. steel channels which rested flange down. Brick in the bottom course were placed on a channel and the mortar slushed into the joints. A thick layer of mortar was then spread over this course and the stirrups and longitudinal steel were pressed into place. The bars were then covered with additional mortar and the three upper courses of brick were laid. These were surmounted by a ½-in. mortar cap which served to cover the tops of the stirrups. This mortar cap was a source of weakness in several of the beams tested owing to its relatively low strength. With the exception of the joint containing the longitudinal steel, the joint thickness was ½ in. The joint carrying the steel was about ½ in. thicker than the diameter of the longitudinal bars.

In the fabrication of the first two beams, C7 and C8, the mason followed the common practice of grooving the joints. This resulted in a weakness in bond and these beams had low strengths. Therefore especial attention was given to filling completely every joint with mortar and much better results were obatined.

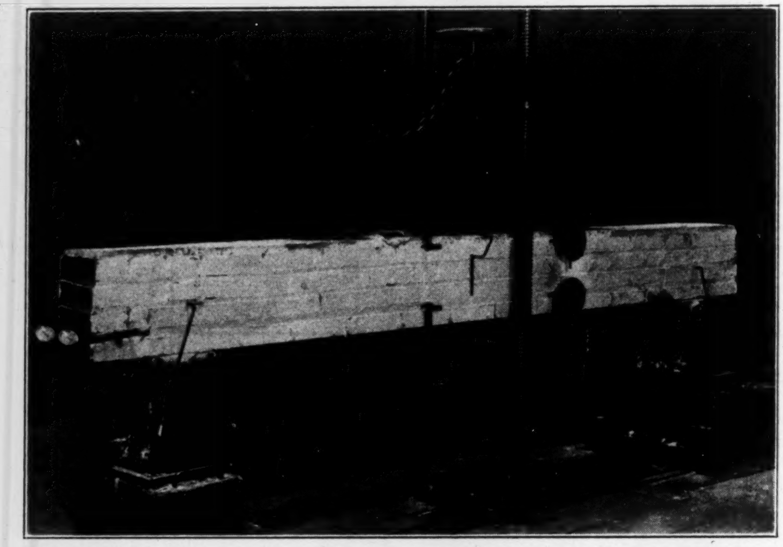


FIG. 2.—Set-up for Testing Beams.

In the fabrication of the earlier made beams of Chicago brick and some of those made of Waupaca brick, the coursing was irregular. Subsequent tests showed that headers were a source of weakness if placed in the top course and contributed to lowering the strengths of beams C5, C6, and C8. The coursing finally used in the remainder of the tests is shown in Fig. 1.

Tests of beams C7 and C8 served to demonstrate that Z-shaped stirrups with the direction of the outstanding legs staggered, though somewhat more difficult to install, could be more effectively spaced than U-stirrups in beams of the size tested. Furthermore, with the coursing used, it was impossible to space the U-stirrups closer than

8 in. This spacing was too great for beams of the depth tested. Hence, the Z-type of stirrup was used as desired throughout the remainder of the tests. Where high percentages of stirrup reinforcement were wanted, Z-stirrups were placed in pairs with the stirrups of a pair separated about one inch.

Since the air of the laboratory in which the beams were made was unusually dry, they were covered with wet burlap kept under canvas until tested in order to provide proper curing conditions for the mortar joints. In order to aid in the detection of cracks the sides of the beams were coated with a white cement grout just before testing. Although the stirrups were not wired to the rods in these tests, the mason and his helper after some practice built 7 beams and 2 piers containing a total of 876 brick in 10 hours.

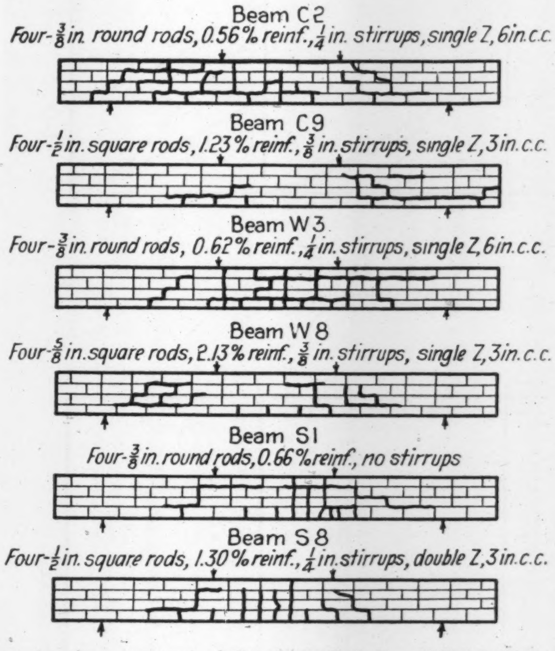
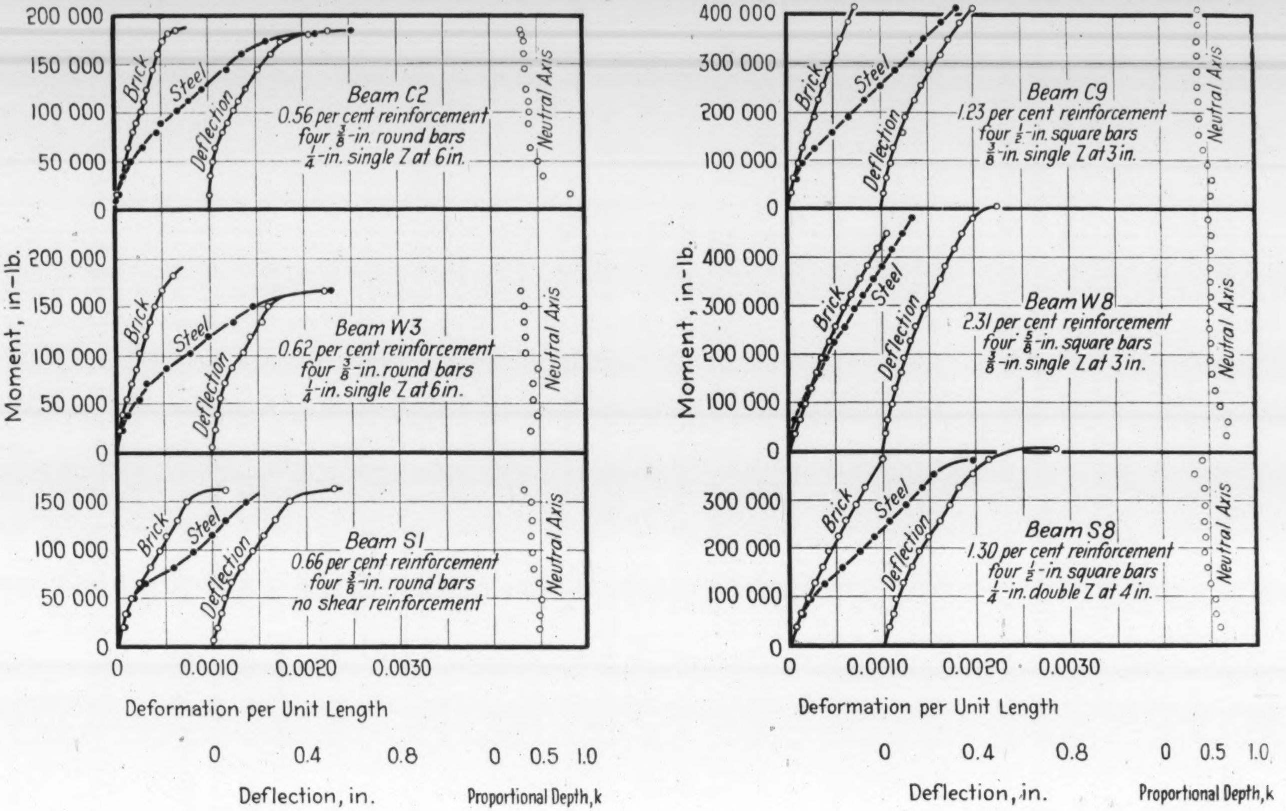

Beam C2
Four-$\frac{3}{8}$ in. round rods, 0.56% reinf., $\frac{1}{4}$ in. stirrups, single Z, 6 in. c.c.
Beam C9
Four-$\frac{1}{2}$ in. square rods, 1.23% reinf., $\frac{3}{8}$ in. stirrups, single Z, 3 in. c.c.
Beam W3
Four-$\frac{3}{8}$ in. round rods, 0.62% reinf., $\frac{1}{4}$ in. stirrups, single Z, 6 in. c.c.
Beam W8
Four-$\frac{5}{8}$ in. square rods, 2.13% reinf., $\frac{3}{8}$ in. stirrups, single Z, 3 in. c.c.
Beam S1
Four-$\frac{3}{8}$ in. round rods, 0.66% reinf., no stirrups
Beam S8
Four-$\frac{1}{2}$ in. square rods, 1.30% reinf., $\frac{1}{4}$ in. stirrups, double Z, 3 in. c.c.

FIG. 3.—Arrangement of Cracks in Reinforced Brick Beams.

## TESTING OF BEAMS

The set-up for testing the beams is shown in Fig. 2. Loads were applied at the third points of an 8-ft. span. Deflections at mid-span were read to 0.01 in. by the wire-mirror-scale deflectometer. Deformeter readings on the top fiber of the brick and in the plane of the longitudinal rods were taken over a 25-in. gage by means of



FIGS. 4 and 5.—Typical Curves Showing Relationship of Applied Bending Moment to the Deflections and to Deformations in the Brick and in the Steel.

wire-wound dials reading to 0.0001 in. as shown in the figure. The Ames dials at the left end of the beam were used to determine slippage of the bars. Loads were slowly applied in increments of $\frac{1}{8}$ to $\frac{1}{12}$ of the ultimate.

*Initial Cracks:*

Vertical tension cracks at mortar joints appeared at loads between 0.25 and 0.5 maximum load. The corresponding strain readings indicated steel stresses of 5000 to 8500 lb. per sq. in. Figure 3 shows sketches of cracked beams which are typical. In the tests of the beams made of Streator brick, snapping sounds, probably due to the cracking of webs of cells, began at loads of 0.37 to 0.56 of the ultimate. Evidence of the security of the bond between the bottom course of brick and the beam was furnished by the fact that none of the bricks in that course dropped off during any test until after the maximum load had been applied. The surprising toughness of these beams after testing was demonstrated by the difficulty experienced in breaking them into small fragments with a sledge.

TABLE V.—A COMPARISON OF STRESSES IN STEEL COMPUTED FROM STRAIN WITH VALUES COMPUTED FROM MOMENTS AT ONE THIRD MAXIMUM LOAD.

Stresses Calculated by Straight-Line Formulas.

| Beam | Steel, per cent | One-half Applied Load Considered, lb. | Proportional Depth of Neutral Axis | | Stress in Steel, lb. per sq. in. | |
|---|---|---|---|---|---|---|
| | | | From Strain | From Calculation | From Strain | From Moment |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* |
| C1 | 0.57 | 1932 | 0.34 | 0.32 | 9 600 | 17 200 |
| C2 | 0.56 | 2010 | 0.36 | 0.31 | 8 100 | 16 900 |
| C4 | 0.61 | 1498 | 0.36 | 0.33 | 6 300 | 14 100 |
| C5 | 0.93 | 2730 | 0.48 | 0.39 | 8 400 | 15 800 |
| C6 | 0.93 | 2820 | 0.42 | 0.38 | 8 100 | 15 600 |
| C9 | 1.23 | 4297 | 0.38 | 0.42 | 10 500 | 16 600 |
| C3 | 0.62 | 1667 | 0.40 | 0.33 | 12 600 | 15 900 |
| C7 | 1.33 | 2583 | 0.57 | 0.44 | 8 400 | 11 200 |
| C8 | 1.24 | 2650 | 0.57 | 0.43 | 4 800 | 11 200 |
| W3 | 0.62 | 1900 | 0.35 | 0.28 | 6 900 | 17 200 |
| W4 | 1.00 | 2750 | 0.44 | 0.34 | 9 000 | 14 800 |
| W5 | 1.04 | 2630 | 0.37 | 0.34 | 13 500 | 15 300 |
| W6 | 1.02 | 2783 | 0.46 | 0.34 | 11 100 | 15 400 |
| W7 | 1.41 | 3928 | 0.39 | 0.39 | 12 600 | 16 400 |
| W8 | 2.31 | 5250 | 0.48 | 0.46 | 9 900 | 14 000 |
| W1 | 0.66 | 1830 | 0.48 | 0.28 | 9 900 | 17 500 |
| W2 | 0.70 | 1470 | 0.41 | 0.29 | 6 900 | 14 300 |
| S1 | 0.66 | 1683 | 0.42 | 0.35 | 6 300 | 17 000 |
| S2 | 0.66 | 2015 | 0.33 | 0.35 | 11 400 | 20 100 |
| S3 | 1.15 | 2500 | 0.52 | 0.43 | 10 200 | 14 400 |
| S4 | 1.29 | 3930 | 0.44 | 0.45 | 12 600 | 16 600 |
| S5 | 1.06 | 2670 | 0.40 | 0.42 | 9 000 | 14 200 |
| S6 | 1.03 | 2860 | 0.44 | 0.42 | 9 900 | 14 900 |
| S8 | 1.30 | 4216 | 0.45 | 0.45 | 13 200 | 17 800 |
| S7 | 2.22 | 4320 | 0.57 | 0.54 | 6 900 | 11 500 |

## RESULTS OF TESTS

*Stresses at Working Loads:*

Typical curves representing the relationship of the applied bending moment to the strains in the brick and in the steel, to the deflection, and to the position of the neutral axis are shown in Figs. 4 and 5.

In Table V is given a comparison of the position of the neutral axis at one third the maximum load computed from deformeter readings of strain with values calculated from the straight-line relation used in reinforced concrete beam design for the position of the neutral axis:

$$k = \sqrt{2pn + (pn)^2} - pn \quad (1)$$

The steel stresses calculated from moment were obtained from the same relation by the formula:

$$f_s = \frac{M}{pjbd^2} \quad (2)$$

Symbols used in the above and in succeeding formulas have the following significance:

$A$ = area of cross-section of longitudinal steel,
$A_s$ = area of cross-section of stirrups per spacing interval, $z$,
$b$ = breadth of beam,
$d$ = effective depth from top of beam to center of rods,
$E_b$, $E_s$ = modulus of elasticity of brick and steel, respectively,
$f_b$, $f_s$ = unit stress in brick and unit stress in steel,
$f_v$ = unit stress in stirrups,
$k$ = ratio of depth of neutral axis from top of beam to $d$,
$j = 1 - \frac{1}{3} k$ for straight-line formula, $1 - \frac{3}{8} k$ for parabolic formula,
$M_b$ = resisting moment in terms of stress in brick,
$M_s$ = resisting moment in terms of stress in steel,
$M$ = bending moment,
$n = E_s/E_b$,
$o$ = perimeter of one longitudinal steel rod,
$p = A/bd$,
$r = A_s/bz$,
$u$ = unit bond stress of mortar to steel, and
$v$ = unit shear stress due to total shear, $V$.

Omitting beams C7 and C8 which were poorly bonded and W1 and W2 which had no stirrups, the values of $k$ determined by strain and by calculation are in fair agreement. The steel stresses computed from strain are much less than those calculated from moment due to the fact that there was considerable tension carried by portions of the brick masonry at the uncracked sections whereas in the stress computation based on moment none is assumed to be taken by the brick.

## TABLE VI.—A COMPARISON OF ULTIMATE VALUES OF $k$, ALSO MAXIMUM MOMENTS.

| Beam | Steel, per cent | Maximum Load, lb. | Proportional Depth of Neutral Axis | | Maximum Moment, in-lb. | | | Type of Failure[d] |
|---|---|---|---|---|---|---|---|---|
| | | | From Strains, $k_1$ | From Calculations, $k_2$[a] | Actual | Computed from $k_2$[b] $M_b$ | Computed from $k_2$[c] $M_s$ | |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* | *8* | *9* |
| C1 | 0.57 | 12 680 | 0.34 | 0.37 | 196 580 | 403 000 | 174 000 | T |
| C2 | 0.56 | 13 120 | 0.36 | 0.37 | 203 800 | 432 000 | 185 000 | T |
| C4 | 0.61 | 12 000 | 0.36 | 0.38 | 186 160 | 366 000 | 166 000 | T |
| C5 | 0.98 | 18 000 | 0.48 | 0.45 | 281 000 | 436 000 | 253 000 | C |
| C6 | 0.93 | 18 000 | 0.42 | 0.44 | 281 680 | 479 000 | 269 000 | T |
| C9 | 1.23 | 26 900 | 0.38 | 0.49 | 426 340 | 562 000 | 405 000 | T+DT |
| W3 | 0.62 | 12 000 | 0.35 | 0.33 | 192 760 | 345 000 | 172 000 | T |
| W4 | 1.00 | 17 500 | 0.44 | 0.39 | 274 260 | 432 000 | 274 000 | T |
| W5 | 1.05 | 16 720 | 0.37 | 0.40 | 262 120 | 393 000 | 256 000 | T |
| W6 | 1.02 | 17 700 | 0.46 | 0.40 | 277 460 | 417 000 | 267 000 | T |
| W7 | 1.41 | 24 180 | 0.39 | 0.45 | 387 260 | 447 000 | 374 000 | C |
| W8 | 2.31 | 32 600 | 0.48 | 0.53 | 515 170 | 499 000 | 475 000 | C |
| S1 | 0.66 | 11 100 | 0.42 | 0.41 | 170 960 | 262 000 | 154 000 | T |
| S2 | 0.66 | 13 000 | 0.33 | 0.41 | 202 950 | 266 000 | 155 000 | T |
| S3 | 1.15 | 16 000 | 0.52 | 0.50 | 250 260 | 320 000 | 216 000 | T |
| S4 | 1.29 | 24 600 | 0.44 | 0.52 | 387 960 | 404 000 | 366 000 | T |
| S5 | 1.06 | 17 000 | 0.40 | 0.49 | 266 260 | 366 000 | 234 000 | T |
| S6 | 1.03 | 18 200 | 0.44 | 0.48 | 285 360 | 378 000 | 240 000 | T |
| S8 | 1.30 | 26 300 | 0.45 | 0.52 | 415 260 | 401 000 | 370 000 | T |

[a] Calculated from parabolic formula.

[b] Values of $f_b$ for Chicago brick 2690, for Waupaca brick 2870, for Streator brick 2090 lb. per sq. in.

[c] Values of the yield point of the steel used in calculating these moments may be found in Table VII.

[d] T=tension; C=compression; DT=diagonal tension.

## TABLE VII.—FIBER STRESSES CALCULATED AT MAXIMUM LOADS SUPPORTED BY REINFORCED BRICK BEAMS.

$S$ = Ultimate compressive strength of brick prisms:
For Chicago brick, $S = 2690$; for Waupaca brick, $S = 2870$;
For Streator brick, $S = 2090$ lb. per sq. in.
Parabolic formulas used for calculating $k$ and stresses from moments

| Beam | Steel, per cent | Proportional Depth of Neutral Axis, $k$ | Maximum Moment, in-lb. | Yield Point of Steel, lb. per sq. in. | Stresses, lb. per sq. in. | | | $\frac{B}{S}$ | Kind of Failure[a] |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Steel from Moment | Steel from Strain | Brick from Moment, $B$ | | |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* | *8* | *9* | *10* |
| C1 | 0.57 | 0.37 | 196 580 | 50 500 | 56 900 | beyond $S_y$ | 1330 | 0.50 | T |
| C2 | 0.56 | 0.37 | 203 800 | 50 500 | 55 600 | beyond $S_y$ | 1270 | 0.47 | T |
| C4 | 0.61 | 0.38 | 186 160 | 50 500 | 57 000 | beyond $S_y$ | 1370 | 0.51 | T |
| C5 | 0.98 | 0.45 | 281 800 | 48 100 | 53 300 | beyond $S_y$ | 1740 | 0.65 | C |
| C6 | 0.93 | 0.45 | 281 680 | 48 100 | 50 300 | beyond $S_y$ | 1580 | 0.59 | T |
| C9 | 1.23 | 0.49 | 426 340 | 51 200 | 54 100 | beyond $S_y$ | 2040 | 0.76 | T+DT |
| C3 | 0.62 | 0.38 | 170 260 | 50 500 | 53 100 | 39 000 | 1290 | 0.48 | DT |
| C7 | 1.33 | 0.50 | 259 160 | 51 200 | 36 900 | 34 000 | 1470 | 0.55 | DT |
| C8 | 1.24 | 0.48 | 266 560 | 51 200 | 36 800 | 22 800 | 1400 | 0.52 | DT |
| W3 | 0.62 | 0.38 | 192 760 | 50 500 | 56 500 | beyond $S_y$ | 1610 | 0.56 | T |
| W4 | 1.00 | 0.39 | 274 260 | 48 100 | 48 100 | beyond $S_y$ | 1840 | 0.64 | T |
| W5 | 1.04 | 0.40 | 262 120 | 48 100 | 49 400 | beyond $S_y$ | 1920 | 0.67 | T |
| W6 | 1.02 | 0.40 | 277 460 | 48 100 | 49 500 | beyond $S_y$ | 1910 | 0.67 | T |
| W7 | 1.41 | 0.45 | 387 260 | 51 200 | 52 800 | beyond $S_y$ | 2490 | 0.87 | C |
| W8 | 2.31 | 0.53 | 515 170 | 41 700 | 45 300 | 39 700 | 2970 | 1.03 | C |
| W1 | 0.66 | 0.34 | 186 260 | 50 500 | 57 800 | beyond $S_y$ | 1710 | 0.60 | T+DT |
| W2 | 0.70 | 0.34 | 183 760 | 50 500 | 57 800 | 39 300 | 1770 | 0.62 | DT |
| S1 | 0.66 | 0.41 | 170 960 | 50 500 | 56 000 | beyond $S_y$ | 1360 | 0.65 | T |
| S2 | 0.66 | 0.41 | 202 950 | 50 500 | 65 700 | beyond $S_y$ | 1600 | 0.77 | T |
| S3 | 1.15 | 0.50 | 250 260 | 41 000 | 47 200 | beyond $S_y$ | 1640 | 0.79 | T |
| S4 | 1.29 | 0.52 | 387 960 | 51 200 | 54 000 | 52 100 | 2020 | 0.97 | T |
| S5 | 1.06 | 0.49 | 266 260 | 41 000 | 46 800 | beyond $S_y$ | 1530 | 0.73 | T |
| S6 | 1.03 | 0.48 | 285 360 | 41 000 | 48 800 | beyond $S_y$ | 1580 | 0.76 | T |
| S8 | 1.30 | 0.52 | 415 260 | 51 200 | 57 600 | beyond $S_y$ | 2170 | 1.04 | T |
| S7 | 2.22 | 0.61 | 426 320 | 41 700 | 37 800 | 32 000 | 2050 | 0.98 | DT |

[a] T=tension; C=compression; DT=diagonal tension.

*Moments and Stresses at Maximum Loads:*

In Table VI the beams which failed by diagonal tension have been omitted and the values of $k_1$ based on strains at or near the ultimate loads are compared with values of $k_2$ computed from the following formula:

$$k_2 = \sqrt{3pn + (\tfrac{3}{2}\,pn)^2} - \tfrac{3}{2}\,pn \dots\dots\dots\dots \quad (3)$$

Also in columns 6, 7 and 8 of the table the values of the actual maximum bending moment are compared with the maximum resisting moments calculated from the parabolic formulas (4) and (5):

TABLE VIII.—VALUES OF MAXIMUM BENDING MOMENTS AND SHEARING STRESSES DEVELOPED BY REINFORCED BRICK BEAMS.

| Beam | Breadth($b$), in. | Effective Depth ($d$), in. | Steel, per cent | Kind of Failure[b] | Total Maximum Load, lb.[c] | Maximum Moment, in-lb.[c] | $\frac{M}{bd^2}$ lb. per sq. in. | $v = \frac{V}{jdb}$,[d] lb. per sq. in. |
|---|---|---|---|---|---|---|---|---|
| *1* | *2* | *3* | *4* | *5* | *6* | *7* | *8* | *9* |
| C1 | 8.30 | 9.20 | 0.57 | T | 12 680 | 196 580 | 278 | 95 |
| C2 | 7.85 | 9.83 | 0.56 | T | 13 120 | 203 800 | 268 | 96 |
| C4 | 8.20 | 8.75 | 0.61 | T | 12 000 | 186 160 | 297 | 96 |
| C5 | 8.20 | 8.88 | 0.98 | C | 18 000 | 281 800 | 435 | 148 |
| C6 | 8.10 | 9.40 | 0.93 | T | 18 000 | 281 680 | 393 | 140 |
| C9 | 7.92 | 9.94 | 1.23 | T+DT | 26 900 | 426 340 | 543 | 208 |
| C3[a] | 8.00 | 8.70 | 0.62 | DT | 11 000 | 170 260 | 281 | 91 |
| C7[a] | 8.05 | 9.00 | 1.33 | DT | 16 600 | 259 160 | 397 | 140 |
| C8[a] | 8.45 | 9.20 | 1.24 | DT | 17 100 | 266 560 | 373 | 133 |
| W3 | 7.70 | 9.03 | 0.62 | T | 12 000 | 192 750 | 307 | 97 |
| W4 | 7.53 | 9.43 | 1.00 | T | 17 500 | 274 260 | 410 | 144 |
| W5 | 7.70 | 8.85 | 1.04 | T | 16 720 | 262 120 | 435 | 143 |
| W6 | 7.56 | 9.20 | 1.02 | T | 17 700 | 277 460 | 433 | 148 |
| W7 | 7.45 | 9.17 | 1.41 | C | 24 180 | 387 260 | 618 | 212 |
| W8 | 7.75 | 8.90 | 2.31 | C | 32 600 | 515 170 | 840 | 294 |
| W1[a] | 7.66 | 8.53 | 0.66 | T+DT | 11 500 | 186 260 | 334 | 100 |
| W2[a] | 7.36 | 8.40 | 0.70 | DT | 11 840 | 183 760 | 353 | 109 |
| S1 | 7.96 | 8.28 | 0.66 | T | 11 100 | 170 960 | 313 | 98 |
| S2 | 7.89 | 8.37 | 0.66 | T | 13 000 | 202 950 | 367 | 115 |
| S3 | 7.95 | 8.43 | 1.15 | T | 16 000 | 250 260 | 443 | 146 |
| S4 | 8.10 | 9.24 | 1.29 | T | 24 600 | 387 960 | 561 | 203 |
| S5 | 8.00 | 9.07 | 1.06 | T | 17 000 | 266 260 | 405 | 142 |
| S6 | 8.07 | 9.26 | 1.03 | T | 18 200 | 285 360 | 411 | 148 |
| S8 | 7.95 | 9.30 | 1.30 | T | 26 300 | 415 260 | 604 | 219 |
| S7[a] | 7.83 | 9.17 | 2.22 | DT | 27 000 | 426 320 | 647 | 243 |

[a] Insufficient stirrups.
[b] T=tension; C=compression; DT=diagonal tension.
[c] Includes weight of beam, 720 to 1020 lb.
[d] Calculations for $j$ made by parabolic formula.

$$M_b = \tfrac{2}{3}\,jkf_b bd^2 \dots\dots\dots\dots \quad (4)$$

$$M_s = pjf_s bd^2 \dots\dots\dots\dots \quad (5)$$

The latter comparisons show that every beam actually exhibited greater moment capacity than the lower result obtained from the formulas.

The results listed in columns 5, 6, 7, and 8 of Table VIII indicate that with the percentages of longitudinal steel used and an adequate number of stirrups it was possible to develop the full fiber strength of these beams. The bond strengths developed in the double shear tests was a rough measure of the shear strength, $v$, of beams having no stirrup reinforcement. The data show that without stirrups it is possible to stress the longitudinal steel to its yield point prior to failure if 0.57 per cent of steel of intermediate grade is used with Chicago brick, 0.68 per cent with Waupaca brick, or 0.95 per cent with the cell-perforated Streator brick. By using adequate percentages of longitudinal steel and stirrups the full compressive strengths of both the Waupaca and the Streator brick masonry were developed.

TABLE IX.—STRESSES IN STIRRUPS, BOND STRESSES, AND DEFLECTIONS OF REINFORCED BRICK BEAMS.

| Beam | Stirrup Ratio, $r$ | Stresses at Maximum Load, lb. per sq. in. | | Deflection Due to an Applied Load Equal to $\frac{1}{4}$ the Maximum, in. | | $\frac{D_o}{D_c}$ |
|---|---|---|---|---|---|---|
| | | Stirrups, $f_v = \frac{v - f_b{}^a}{r}$ | Bond, $u = \frac{vb}{\Sigma o}$ | Observed, $D_o$ | Calculated, $D_c$ | |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* |
| C1 | 0.00094 | 5 300 | 168 | 0.021 | 0.033 | 0.64 |
| C2 | 0.00100 | 6 000 | 161 | 0.023 | 0.031 | 0.77 |
| C4 | no stirrups | .... | 168 | 0.020 | 0.035 | 0.57 |
| C5 | 0.00381 | 15 200 | 202 | 0.055 | 0.047 | 1.17 |
| C6 | 0.00386 | 12 900 | 189 | 0.046 | 0.041 | 1.12 |
| C9 | 0.00455 | 25 900 | 210 | 0.044 | 0.048 | 0.91 |
| C3 | no stirrups | .... | 156 | 0.065 | 0.034 | 1.91 |
| C7 | 0.00336 | 14 900 | 143 | 0.065 | 0.039 | 1.67 |
| C8 | 0.00428 | 10 100 | 143 | 0.034 | 0.037 | 0.92 |
| | | | | | | 1.08 avg. |
| W3 | 0.00102 | 0 | 160 | 0.070 | 0.037 | 1.89 |
| W4 | 0.00208 | 21 100 | 181 | 0.047 | 0.026 | 1.81 |
| W5 | 0.00305 | 14 100 | 184 | 0.052 | 0.034 | 1.53 |
| W6 | 0.00310 | 15 400 | 187 | 0.041 | 0.033 | 1.24 |
| W7 | 0.00485 | 23 200 | 201 | 0.079 | 0.043 | 1.84 |
| W8 | 0.00467 | 41 800 | 226 | 0.062 | 0.054 | 1.15 |
| W1 | no stirrups | .... | 164 | 0.060 | 0.028 | 2.14 |
| W2 | no stirrups | .... | 172 | 0.048 | 0.032 | 1.50 |
| | | | | | | 1.64 avg. |
| S1 | no stirrups | .... | 167 | 0.048 | 0.048 | 1.00 |
| S2 | no stirrups | .... | 194 | 0.044 | 0.051 | 0.86 |
| S3 | no stirrups | .... | 186 | 0.060 | 0.054 | 1.10 |
| S4 | 0.00446 | 18 700 | 209 | 0.062 | 0.060 | 1.03 |
| S5 | 0.00293 | 7 500 | 182 | 0.051 | 0.047 | 1.08 |
| S6 | 0.00194 | 1 400 | 191 | 0.040 | 0.047 | 0.85 |
| S8 | 0.00295 | 33 700 | 222 | 0.070 | 0.059 | 1.19 |
| S7 | 0.00462 | 26 800 | 188 | 0.058 | 0.064 | 0.91 |
| | | | | | | 1.00 avg. |

[a] $f_b$ = 90 lb. per sq. in. for Chicago brick, 100 lb. per sq. in. for Waupaca brick, 120 lb. per sq. in. for Streator brick.

*Coefficients of Resistance and Shear Stresses:*

In Table VIII values of the maximum coefficients of resistance, $\frac{M}{bd^2}$, and maximum unit shear stresses $v$ are tabulated. These are useful coefficients for design purposes. Besides the latter significance, the high values of $\frac{M}{bd^2}$ and $v$ developed in the tests of beams C9, W7, W8, S4, S7, and S8 furnish confirmation of the flexural strength possibilities of reinforced brick beams.

*Stirrup Stresses:*

The value of the Z-stirrups in the stronger beams C9, W8, S8, and S7 is shown by the high tensile stresses listed for the stirrups of these beams in column 3 of Table IX. The diagonal tension failure in beam S7 was apparently due to weakness in mortar bond at the joint carrying the longitudinal rods.

*Bond Stresses:*

In six of these beams, bond stresses between the mortar and the deformed bars exceeded 200 lb. per sq. in. as shown in column 4, Table IX. There was no evidence from the readings of the Ames dials of slippage of rods during these tests.

*Deflections:*

Observed mid-span deflections, $D_o$, at an applied load equal to one-fourth of the maximum have been compared with computed deflections, $D_c$, in column 7 of Table IX. The computed deflections were based on the formula:[1]

$$D_c = \frac{23}{1296} \frac{\alpha P l^3}{E_b b d^3} \quad \text{.............................} \quad (6)$$

where $P$ is the total applied load equal to one fourth the ultimate and $\alpha = 12\ (1 + pn) \div (1 + 4\ pn)$. This formula assumes tension to be taken by the brickwork below the neutral axis but makes no allowance for deflection due to shear. The agreement between the calculated and observed deflections is poor for these beams. Some of the discrepancies are doubtless due to the smallness of the observed values and the fact that the readings were taken only to 0.01 in.

Tests were recently made at the U. S. Bureau of Standards[2] on six 12 by 14-in. beams carrying 1 per cent reinforcement and having coursing similar to the above. The span in those tests was 12 ft. and the loads were applied at the quarter points. Three of the beams were made of Chicago brick and three of Philadelphia brick. For loads of one fourth the ultimate, calculations for these beams give an average ratio $D_o/D_c$ of 0.89 with maximum departures of 12 per cent on either side of the average.

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[1] F. E. Turneaure and E. R. Maurer, "Principles of Reinforced Concrete Construction," Fourth Edition, p. 154, John Wiley and Sons, New York City (1932).

[2] B. E. Parsons, A. H. Stang, and J. W. McBurney, "Shear Tests of Reinforced Brick Masonry Beams," U. S. Department of Commerce *Research Paper No. 504*.

## CONCLUSIONS

1. The results of these tests show that it is possible to develop a high degree of flexural strength in reinforced brick beams.

2. With proper design of stirrup and longitudinal reinforcement, coefficients of resistance, $M/bd^2$, in excess of 500 lb. per sq. in. and maximum shear stresses, $v$, in excess of 200 lb. per sq. in. were obtained with all three varieties of brick. The maximum values of $M/bd^2$ and $v$ were 840 and 294 lb. per sq. in., respectively.

3. Where web reinforcement is required and no longitudinal rods are bent up, a stirrup spacing approximately one half the effective depth of the beam is satisfactory.

4. The tests indicate that the formulas used in the calculation of fiber, shear, and bond stresses and deflections for reinforced concrete beams can, with proper constants, be used in like calculations for reinforced brick beams.

5. In order to secure good strengths *particular attention must be given to see that all joints are completely filled.*

6. In designing the arrangement of coursing, it is preferable to eliminate headers from heavily compressed portions of beams.

7. The 1 lime:3 cement:12 sand mortar used in these experiments gave very satisfcatory results for this class of work.

8. With the mortar used in these tests, shear strengths were developed without stirrups of 93, 104, 146 lb. per sq. in. in the beams of Chicago, Waupaca and Streator brick, respectively.

9. Brick of the Chicago type when dry should be sprinkled prior to laying to secure best results.

10. A knowledge of the moisture content desirable in a given brick and of the bond of the mortar to it is essential to proper design of reinforced brick beams.

11. Although conditions in these tests were not ideal for efficient bricklaying, the rate of operation indicated that with proper design of reinforcement excellent speeds can be attained in laying reinforced brick beams.

12. Among the more important problems in reinforced brick beam construction which should receive thorough study are the effect of duration of load on strength and stiffness, the resistance of such beams to freezing and thawing and to fire, the development of practical types of web reinforcement, and the utilization of thin keyed tile in place of the bottom course of brick.

# DISCUSSION

MR. F. O. ANDEREGG.[1]—Because of the difficulty of getting good workmanship, the danger of corrosion of the reinforcing rods in brick beams exposed to the weather, or in capillary contact with masonry walls exposed to the weather, is sufficient to warrant galvanizing or cadmium plating. The accompanying Fig. 1 shows badly rusted steel used to support an overhanging cornice after 27 years of life. *A* and *B* are fabricated of angle irons. Two rods *C* had extended from the channel iron *D* into hole *E* of the cornice unit, but their ends had been completely rusted off. In a few places the mortar still affords protection to the steel.

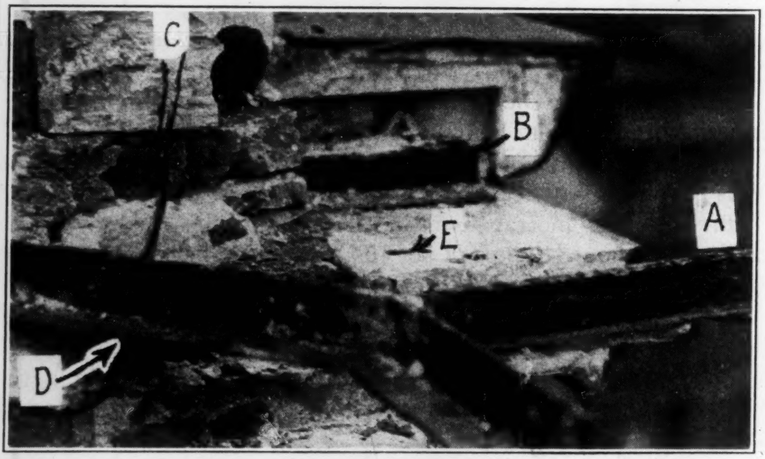

FIG. 1.—Badly Rusted Steel in Overhanging Cornice.

In the construction of brick beams I have been using a different principle. By putting the under side of the beam where it is weakest, under an initial stress of, say 5000 lb. per sq. in., in the direction opposite to that applied when loaded, it is possible to take advantage of the high compressive strength of burned clay units, and construct beams of light weight but of good load-carrying capacity. A combination of well-burned units, high quality steel, and high quality mortar, thoroughly compacted and well compressed in thin joints, has produced in a series of beams, at 48 hr. after building, a stress in the extreme fiber more than twice as great as the stress initially applied.

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[1] Consulting Specialist on Building Materials, Pittsburgh, Pa.

MR. D. E. PARSONS.[1]—Regarding the estimated strength under conditions just illustrated, it seems to me that one would have to temper the estimate somewhat because of the difference in the strength of clay products when laid in combination with mortar joints and when tested as single units. Ordinarily we do not expect efficiencies of more than 20 to 30 per cent. While the type of construction that is illustrated by Mr. Anderegg may have some possibilities, the estimated useful strength seems a little high.

I was interested in Mr. Withey's remarks regarding the attitude of brick masons. Our experiences at the U. S. Bureau of Standards, where we have had a number of test specimens made by outside bricklayers, have been very similar. The tendency seems to be for the bricklayer to groove the joint, and it requires considerable education before he can be induced to lay the brick without the grooved joints.

MR. HUGO FILIPPI.[2]—I should like to call attention to a test that has been going on now for nearly three years. I have had under observation at a Chicago brickyard, a T beam, rather heavily reinforced, that has been directly exposed to the weather, under load, for a period of about two years and nine months. From time to time I have cut away a brick to see whether or not the reinforcement has been attacked, and so far I have found no such evidence. In each case I have, of course, very carefully painted up the freshly exposed portion so as to prevent penetration of moisture into the beam.

There is on record, the construction of quite a number of reinforced brickwork structures in this country built during the past two or three years. I have examined a number of those structures, using a chisel and hammer, and to date I have found no evidence of steel corrosion.

I can hardly conceive any great danger from corrosion if the bond between the brick and the mortar is sound, and if the mortar mixture is correct. The mortar mix used in recent reinforced brickwork structures has generally consisted of one part portland cement to 15 per cent to 50 per cent of lime, and from 3 to 4 parts of well-graded sand. Such a mortar, if properly prepared and if the brickwork is properly laid, is dense, reasonably impervious, produces a good bond and will satisfactorily protect the steel against corrosion.

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[1] Chief, Masonry Construction Section, U. S. Bureau of Standards, Washington, D. C.

[2] General Superintendent, Illinois Brick Co., Chicago, Ill.

MR. E. E. KRAUSS.[1]—As to the possibility of corrosion of the metal in reinforced brick masonry, there are several points to be considered. The mortar itself must be of good materials, especially clean sand. The use of dirty sand or water containing silt is no more permissible in reinforced brick masonry than in reinforced concrete. The workability of mortar should be secured by the addition of lime and not clay. We all have observed the rusting of reinforcing in poor or porous mortar, be that mortar a part of concrete or of brick masonry.

In 1859 the Georgetown water reservoir at Washington, D. C., was built of reinforced brick masonry. It was a dome-shaped structure 120 ft. in diameter and about 50 ft. high, reinforced with hoop iron having a cross-section of from $\frac{1}{32}$ by $\frac{3}{4}$ in. to $\frac{1}{8}$ by 2 in. laid in $\frac{1}{2}$ in. mortar joints. The structure was in continuous service until 1897. A few years ago it was torn down, when it was my privilege to inspect various sections of wall. No sign of corrosion was visible on the bands. The mortar was made with hydraulic cement. Two large sections of wall with embedded reinforcement, each weighing about one ton, are being preserved at the yard of West Brothers Brick Co., and one similar section at the yard of the United Clay Products Co., all of Washington, D. C. Inspection of these wall sections will prove the absence of rust in new breaks.

I should like to ask Mr. Withey whether, in connection with the physical tests of the brick used in the beams, any effort was made to determine what percentage or rate of absorption would give the maximum strength of mortar bond and shear.

MR. M. O. WITHEY.[2]—We did not go that far into our investigation. We have the time rate of absorption for those bricks. We did not have time to make all of these auxiliary tests, but it would be very desirable, I am sure, in this short series.

MR. KRAUSS.—It might be of general interest to know that in some experiments made to determine the effect of rate of absorption upon mortar strengths, it was found that during the period of absorption, there was a varying rate of absorption. That is, the water entered the brick slowly at first, obtained a maximum rate of entering and then a retarding rate. It was found that a brick saturated to the maximum rate of absorption, made into a brick beam, or a bond test specimen, showed maximum strength. The strength curve followed the rate of absorption curve. This may have some bearing on future work.

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[1] Member of Executive Committee, National Brick Manufacturers' Assn., Philadelphia, Pa.

[2] Professor of Mechanics, University of Wisconsin, Madison, Wis.

MR. W. C. VOSS.[1]—The question raised by Mr. Krauss as to the rate of absorption is an important one. It is not how much is absorbed. The question is, what is the rate at the time the mortar is doing its work. Unless you get a definite attachment of the mortar by chemical action, or otherwise, to the face of the brick, which has a shrinkage very close to that of the brick, you are going to have a break at that point, and this will suck water faster than any brick or mortar you may have used.

MR. WITHEY.—With regard to the rate of absorption, we made tests on these bricks at 5 minutes, 30 minutes, 6 hours, 12 hours, 24 hours, and obtained the time rate of absorption curve. Before laying we wetted the brick that we thought needed wetting. The record of the percentage of absorption at the time the beams were made is given in the paper. I think the brick carried more water than corresponded to the point of maximum pickup mentioned by Mr. Krauss.

I think that we must all recognize that reinforced brickwork is somewhat different from the usual type of brick masonry, and we must not theorize too much on what is going to happen until we have had adequate test data to supplement our theories.

MR. P. M. WOODWORTH.[2]—Mr. Withey in his conclusion No. 7 states that the 1 lime:3 cement:12 sand mortar used in these experiments gave very satisfactory results for this class of work. I wish to call attention to the fact that this proportion is based on the weight of the separate materials.

Expressed in the usual volumetric proportions, as used on the job, the mortar was 1 volume of portland cement, $\frac{3}{4}$ volume of hydrated lime, and $3\frac{3}{4}$ volumes of well-graded sand.

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# PERMEABILITY OF BRICK MASONRY WALLS—AN HYPOTHESIS

### BY W. C. VOSS[1]

## SYNOPSIS

When a brick wall allows the rain to penetrate something is wrong with the selection of materials, the design of the wall, the workmanship or a combination of these factors. The selection of the proper brick with its most advantageous mortar is of great importance. The selection of a dense brick and a dense mortar does not necessarily answer the question. If a wall leaks due to poorly selected materials it is because the brick itself has separated from the mortar and the water passes through these minute cracks.

A thorough study of the properties of a brick to produce the best results seems to indicate that a brick should absorb from 5 to 10 per cent of its weight in water in two days and that it should absorb this water at a low rate after the first 10 or 15 min. This allows the mortar to build up a "bond layer" at the brick line which will insure no separation at that point. Lime is required to supply the materials necessary for this action, and the best general mortar for all general uses with good brick is one composed of one part of portland cement, one part of lime (putty preferably) and from five to six parts of sand, all by volume. This mortar with a brick which has a relatively good suction has given fine results as determined by microscopic examination of the joint and by tests of bond strength.

The architect should consider carefully the details of his wall construction and the contractor should see that his workmen fill all joints thoroughly. Without these precautions even the best mortar may fail to make a tight brick wall.

## LIMITATIONS OF THE PAPER

A careful review of the facts presented by eminent investigators on the general subject of permeability of brick masonry walls would lead us to attribute leaky walls to one or more of the following causes:

1. Properties of the masonry unit and of the mortar separately and also as an assemblage.
2. Detail character of wall and auxiliary construction.
3. Workmanship.

Of these, only the first is considered here, it being remembered, however, that the other two are at least of equal importance, but much more easily controlled, and perhaps less complicated in their scientific aspects.

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Of the many masonry units, which all deserve more careful study at some time, only the solid unit of clay or shale, commonly called the brick, will be discussed, and of the many varieties of such units, only four types are offered for consideration. Likewise, the mortars considered are limited to a few mixes. With this rather specialized curtailment of products we find a group of variables that may well confuse our data, if neglected, misunderstood, uncontrolled or perhaps even unknown.

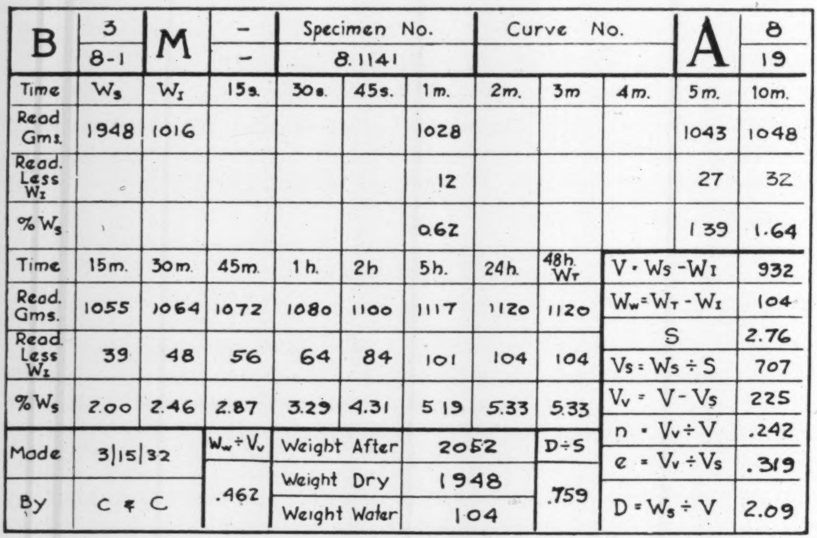
## BRICK CHARACTERISTICS

Clay or shale in its many variations has essentially a plate-like structure with minerals possessing a well-developed basal cleavage. The important minerals composing the clay are usually hydrous silicates and such possess the capacity to attract surface water, thereby separating the various particles. It is for this reason that clays are usually plastic and that they shrink on drying. The last 15 per cent of moisture is relatively hard to drive off and bears an important relation to the structure produced during the burning of brick made from this material.

The size of pores and their inter-connection constitute permeability. These characteristics can and have been controlled, and it is to such control that we must look to produce the desired results. The processing of clays by addition of sand, grog and ground brick, and the control of the plasticity in molding have been found to produce radical differences in the resulting brick even when made of the same general body clay. Sand grains act differently in burning than the plate-like clay particles, and produce shrinkage areas around each grain of sand which may be more or less intercommunicating, depending entirely upon the amount of sand used and the plasticity and composition of the clay itself. Careless use of the run-of-the-pit clay and poor control in burning may well produce wide variations in structure, and it is to the uniformity of a known structure that we must look for the solution of our problem so far as the brick affects it.

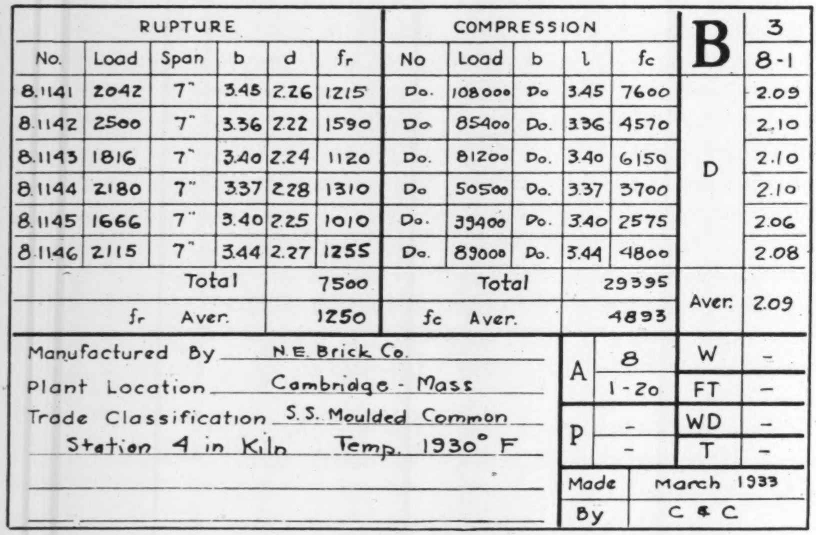
Four brick types have been studied. These are:

1. Dry-press shale face brick (commonly called a pressed brick—laboratory type B1);
2. Wire-cut, water-struck, molded, shale face brick (commonly called wire-cut shale—laboratory type B5);
3. Molded, sand-struck common (N.E. manufacture—laboratory Type B3); and
4. Molded, water-struck common (N.E. manufacture—laboratory Type B4).



| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| B | 3 | M | – | Specimen No. | | | Curve No. | | | A | 8 |
| | 8-1 | | – | 8.1141 | | | | | | | 19 |
| Time | $W_s$ | $W_I$ | 15s. | 30s. | 45s. | 1m. | 2m. | 3m | 4m. | 5m. | 10m. |
| Read Gms. | 1948 | 1016 | | | | 1028 | | | | 1043 | 1048 |
| Read. Less $W_I$ | | | | | | 12 | | | | 27 | 32 |
| % $W_s$ | | | | | | 0.62 | | | | 1 39 | 1.64 |
| Time | 15m. | 30m. | 45m. | 1 h. | 2h | 5h. | 24h. | 48h. $W_T$ | $V = W_s - W_I$ | | 932 |
| Read. Gms. | 1055 | 1064 | 1072 | 1080 | 1100 | 1117 | 1120 | 1120 | $W_w = W_T - W_I$ | | 104 |
| Read. Less $W_I$ | 39 | 48 | 56 | 64 | 84 | 101 | 104 | 104 | S | | 2.76 |
| | | | | | | | | | $V_s = W_s \div S$ | | 707 |
| % $W_s$ | 2.00 | 2.46 | 2.87 | 3.29 | 4.31 | 5 19 | 5.33 | 5.33 | $V_v = V - V_s$ | | 225 |
| Made | 3/15/32 | | $W_w \div V_v$ | Weight After | | 2052 | | $D \div S$ | $n = V_v \div V$ | | .242 |
| | | | | | | | | | $e = V_v \div V_s$ | | .319 |
| By | C & C | | .462 | Weight Dry | | 1948 | | .759 | | | |
| | | | | Weight Water | | 1·04 | | | $D = W_s \div V$ | | 2.09 |

FIG. 1.—Absorption Record for Brick.[1]



| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| RUPTURE | | | | | | COMPRESSION | | | | | B | 3 |
| No. | Load | Span | b | d | $f_r$ | No | Load | b | l | $f_c$ | | 8-1 |
| 8.1141 | 2042 | 7" | 3.45 | 2.26 | 1215 | Do. | 108000 | Do | 3.45 | 7600 | D | 2.09 |
| 8.1142 | 2500 | 7" | 3.36 | 2.22 | 1590 | Do | 85400 | Do. | 3.36 | 4570 | | 2.10 |
| 8.1143 | 1816 | 7" | 3.40 | 2.24 | 1120 | Do. | 81200 | Do. | 3.40 | 6150 | | 2.10 |
| 8.1144 | 2180 | 7" | 3.37 | 2.28 | 1310 | Do | 50500 | Do. | 3.37 | 3700 | | 2.10 |
| 8.1145 | 1666 | 7" | 3.40 | 2.25 | 1010 | Do. | 39400 | Do. | 3.40 | 2575 | | 2.06 |
| 8.1146 | 2115 | 7" | 3.44 | 2.27 | 1255 | Do. | 89000 | Do. | 3.44 | 4800 | | 2.08 |
| | | Total | | | 7500 | | Total | | | 29395 | Aver. | 2.09 |
| | | $f_r$ Aver. | | | 1250 | | $f_c$ Aver. | | | 4893 | | |

Manufactured By N.E. Brick Co.
Plant Location Cambridge - Mass
Trade Classification S.S. Moulded Common
Station 4 in Kiln Temp. 1930° F

| | | | |
|---|---|---|---|
| A | 8 | W | – |
| | 1-20 | FT | – |
| P | – | WD | – |
| | – | T | – |
| Made | March 1933 | | |
| By | C & C | | |

FIG. 2.—Strength Record for Brick.[1]

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[1] These cards are presented as examples of record only, although the data are actual results of ests.

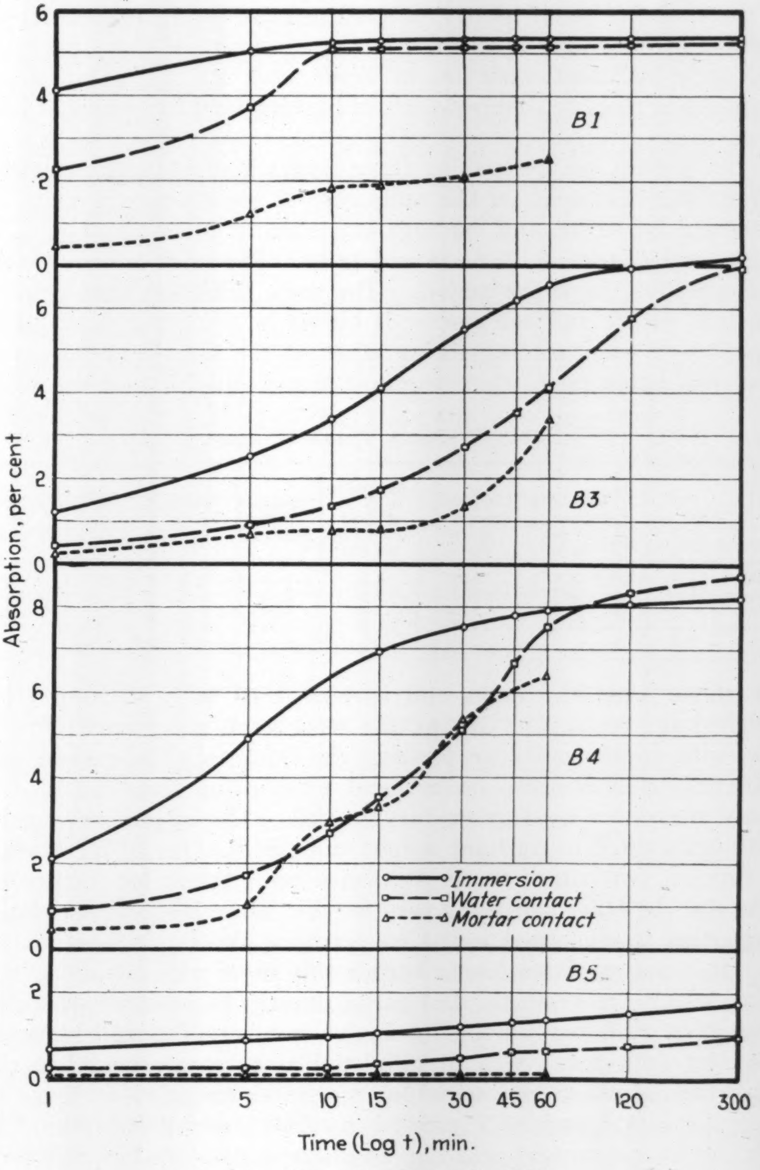


FIG. 3.—Absorption Curves for Brick.

These were selected after a careful study of the rate and amount of absorption of about twenty different manufacturers' brick and were selected because they involved a fair cross-section of the usual types and gave sufficient variation in crude clay structure, processing, pressures in molding, temperatures of burning, and roughness of bond face.

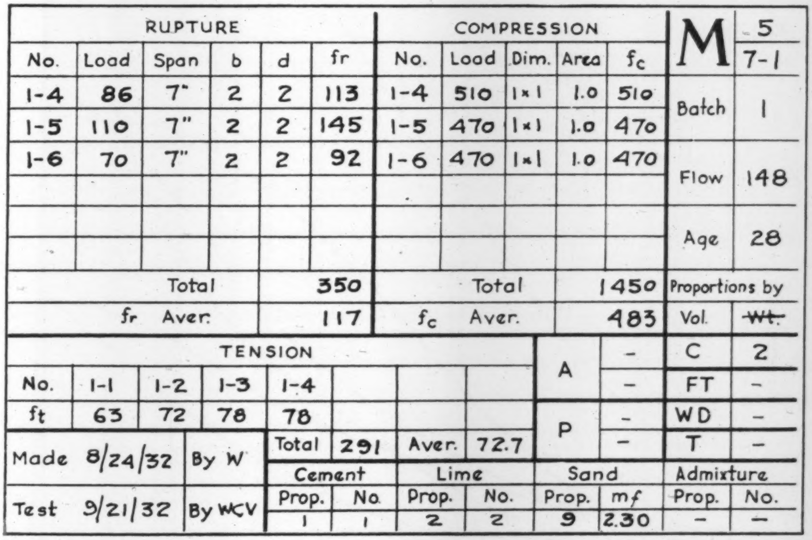
At present we are making three determinations which relate to absorption. Six brick of the same general specific-mass gravity are immersed for 48 hr. and readings are taken at intervals of 1, 5, 10, 15, 30 and 45 min., 1, 2, 5, 24 and 48 hr. The brick are completely immersed for the entire period. The same brick are then dried to constant weight and one surface is placed in contact with water for a period of 5 hr., readings being taken at the same intervals as for total immersion up to that length of time. In this case the bricks are raised from the racks and surface dried with a damp cloth, weighed and replaced. Groups of 6 brick each are placed into contact with fresh mortar by pressing the brick into the mortar with three slight movements under hand pressure of about 10 lb. As many groups are used as mortars studied. The original 6 brick are then again dried to constant weight and are then tested for modulus of rupture, one half of the broken brick being tested for compressive strength, the other half being used for the determinations of chemical properties, specific gravity, and for petrographic examination.

TABLE I.—BRICK CHARACTERISTICS.

| Brick | Modulus of Rupture, $f_r$, lb. per sq. in. | Compressive Strength, $f_c$, lb. per sq. in. | Absorption in 48 hr. of Total Immersion, per cent | Specific-Mass Density, $D$ | Specific Gravity | Void Ratio | Coefficient of Saturation |
|---|---|---|---|---|---|---|---|
| B1.................. | 1635 | 8 035 | 5.50 | 2.16 | 2.69 | 0.252 | 0.579 |
| B3.................. | 1675 | 3 305 | 9.63 | 1.92 | 2.58 | 0.360 | 0.733 |
| B4.................. | 1005 | 5 965 | 7.18 | 1.93 | 2.46 | 0.251 | 0.572 |
| B5.................. | 2915 | 16 475 | 3.31 | 2.24 | 2.41 | 0.191 | 0.501 |

It is not my intention to burden this paper with a mass of such data, which are available, but rather merely to illustrate the characteristics exhibited by the brick. A number of record cards are used for such data, each so tabulated as to insure complete consideration of all known variables. Typical examples of these are shown in Figs. 1 and 2. Figure 1 is used for absorption (either of the brick or of the mortar), and Fig. 2 for strengths.

Considering the four types of brick mentioned above, it will, however, be interesting to note some average results of such tests. Figure 3 illustrates the average rate of absorption of 6 brick of each type by total immersion, surface contact with water, and with mortar (M4). Table I illustrates the average modulus of rupture, compressive strength, percentage of absorption in 48 hr. of total immersion, specific-mass density *D*, specific gravity, void ratio, and coefficient of saturation for each brick type. The coefficient of saturation may be defined as the ratio of the voids filled by total immersion in 48 hr. to the actual voids existent in the brick. This ratio gives an indication of the degree of intercommunication of the capillaries, and perhaps of vitrification.



| RUPTURE | | | | | | COMPRESSION | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| No. | Load | Span | b | d | fr | No. | Load | Dim. | Area | $f_c$ |
| 1-4 | 86 | 7" | 2 | 2 | 113 | 1-4 | 510 | 1×1 | 1.0 | 510 |
| 1-5 | 110 | 7" | 2 | 2 | 145 | 1-5 | 470 | 1×1 | 1.0 | 470 |
| 1-6 | 70 | 7" | 2 | 2 | 92 | 1-6 | 470 | 1×1 | 1.0 | 470 |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| Total | | | | 350 | | Total | | | 1450 | |
| $f_r$ Aver. | | | | 117 | | $f_c$ Aver. | | | 483 | |

| M | 5<br>7-1 |
|---|---|
| Batch | 1 |
| Flow | 148 |
| Age | 28 |
| Proportions by | |
| Vol. | ~~Wt.~~ |
| C | 2 |
| FT | – |
| WD | – |
| T | – |

| TENSION | | | | | | | |
|---|---|---|---|---|---|---|---|
| No. | 1-1 | 1-2 | 1-3 | 1-4 | | | |
| $f_t$ | 63 | 72 | 78 | 78 | | | |
| | | | | Total | 291 | Aver. | 72.7 |

| | |
|---|---|
| A | – |
| | – |
| P | – |
| | – |

| | | | Cement | | Lime | | Sand | | Admixture | |
|---|---|---|---|---|---|---|---|---|---|---|
| Made | 8/24/32 | By W | Prop. | No. | Prop. | No. | Prop. | mf | Prop. | No. |
| Test | 9/21/32 | By WCV | 1 | 1 | 2 | 2 | 9 | 2.30 | – | – |

FIG. 4.—Strength Record for Mortar.[1]

## MORTAR CHARACTERISTICS

In mortar, we again have numerous variables. The cementing material, the aggregate, the amount of water, the shape and size of test specimens, the curing and the subsequent treatment all offer possibilities of infinite data, and of great error in drawing conclusions if any of the variables are unknown, ignored or insufficiently considered. From the point of view of our problem, cementing materials vary widely in their effect. Portland cements, with or without special treatment, produce very different results. Limes, high calcium *versus* dolomitic, with or without treatment, greatly affect the results. The aggregate may be fine and well graded, or coarse and poorly graded. Such variations materially affect results, even though not evident in ordinary strength tests. What may be the most advantageous curing for one mortar may be detrimental to another. The correct application of the results of laboratory tests on a mortar to the behavior of the same mortar used as a bonding agent between known brick may well be a difficult task.

---

[1] This card is presented as an example of record only, although the data are actual results of tests.

Cognizant of all such difficulties, yet appreciating the value which may reside in our old methods, and attempts at new trials in addition, the author has formulated a procedure which is a composite of all. In general, after a study of several brands of portland cement, one brand is used consistently throughout any series. The same holds true for the lime, a high-calcium product, used both as the hydrate and as a putty. The sand has been a clean, coarse, well-graded brick sand with a fineness modulus of from 2.00 to 2.50. All specimens have been tested for tension, compression, modulus of rupture and for absorption. They have been cured in two ways. This will be considered later.

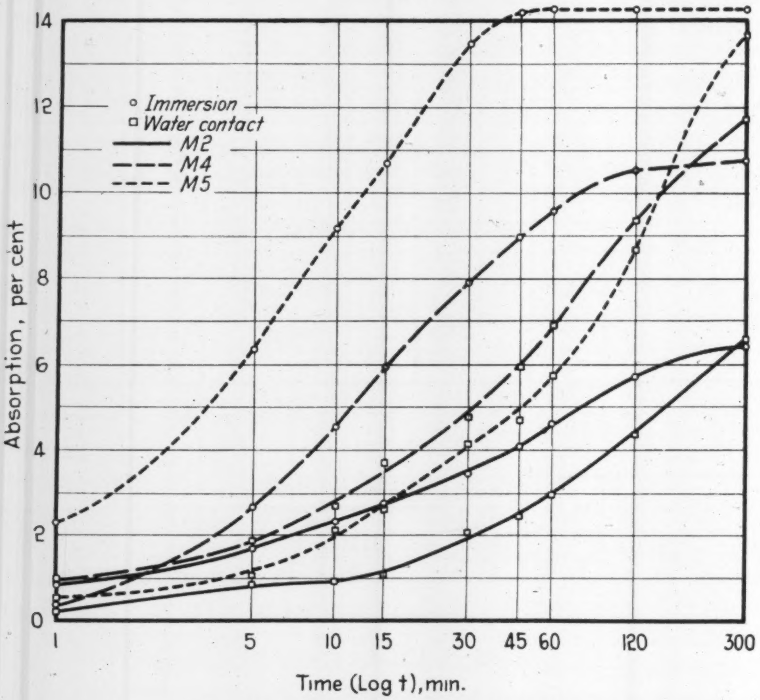


FIG. 5.—Absorption Curves for Mortar.

The mixes used have been widely variant. From a proportion of 1 part of cement to 3 parts of sand without any lime, the lime content has been varied by percentages from 10 per cent by weight of cement to a mortar composed of 1 part by volume of lime and 3 parts of sand without any cement. Patented brick mortars have been used to determine the effect they might have when substituted for the portland cement in the usual mix of 1 cement to 3 sand. Here again a mass of data is available but space prohibits its inclusion here. A few record cases will suffice to illustrate the procedure and the general results indicated. Cards such as shown in Fig. 1 are used for the absorption tests of the mortar specimens and the record card shown in Fig. 4 gives the general strength data for a given mortar. Singling out a group of mixes now most generally advocated we might possibly settle upon these:

1 part of cement, 3 parts of sand, 10 per cent of lime (laboratory type M2)
1 part of cement, 1 part of lime, 6 parts of sand (laboratory type M4)
1 part of cement, 2 parts of lime, 9 parts of sand (laboratory type M5)

Figure 5 illustrates the average rate of absorption for mortar specimens of each type by total immersion and by surface contact.

TABLE II.—MORTAR CHARACTERISTICS.

| Mortar | Flow | Modulus of Rupture, $f_r$ lb. per sq. in. | Compressive Strength, $f_c$, lb. per sq. in. | Tensile Strength, $f_t$, lb. per sq. in. | Absorption in 48 hr. of Total Immersion, per cent | Specific-Mass Density, $D$ | Specific Gravity | Void Ratio | Coefficient of Saturation |
|---|---|---|---|---|---|---|---|---|---|
| M2......... | 150 | 925 | 3185 | 273 | 6.39 | 2.01 | 2.60 | 0.298 | 0.557 |
| M4......... | 148 | 529 | 1214 | 174 | 11.55 | 1.87 | 2.40 | 0.289 | 0.970 |
| M5......... | 148 | 210 | 469 | 84 | 14.53 | 1.73 | 2.48 | 0.428 | 0.845 |

Table II gives the flow, average modulus of rupture, compressive strength, tensile strength, percentage absorption in 48 hr., specific-mass density $D$, specific gravity, void ratio and coefficient of saturation of each of these types, as for the brick.

Much could be said about standard curing methods. There is no question but that the methods now used as standard for cement mortars and concrete are quite ideal for uniformity in these materials, but it places a mortar which is relatively high in lime content at a distinct disadvantage. Until a method can be devised which will give an indication of what may be expected of high-lime mortars at 3 or 6 months, the profession must judge high-lime content mortars in comparison with cement mortars on a strength basis at 28 days. Such a judgment is positively an erroneous one, as strength is not the criterion for mortar bond. Such a study will be very much worth while.

## BRICK AND MORTAR COMBINATIONS

In brick and mortar combinations, we have another group of variables which has been known to affect the permeability of walls in greater or less degree. The conception which grew up in the minds of the profession with the "strength complex" developed by laboratory determinations resulted in the common conclusion that a dense and impervious brick laid in a dense, impervious mortar would produce an impermeable wall. This, of course, has been found to be in error, and for every success which a brick may claim there are also a number of failures. The same holds true for mortars. This would indicate that for a given brick, certain mortars produce better results than others, and *vice-versa*. It is also generally conceded, and was contended by the author many years ago, that water did not pass through the brick or the mortar (where these were of good grade) but between these two, due to a break in the bond.

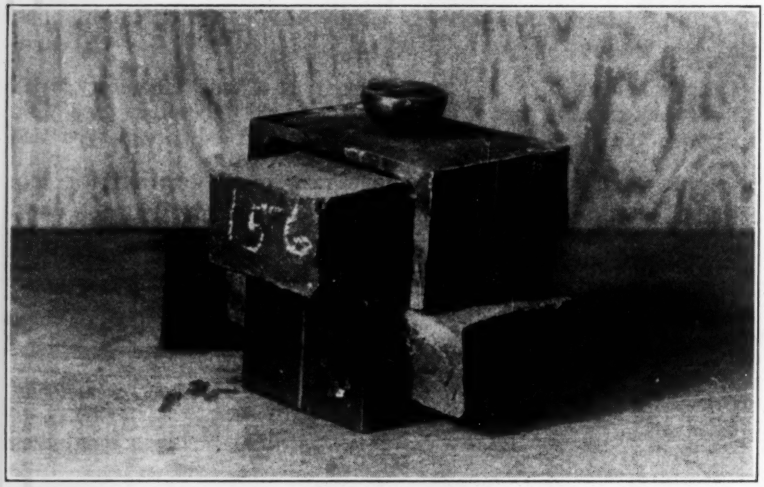
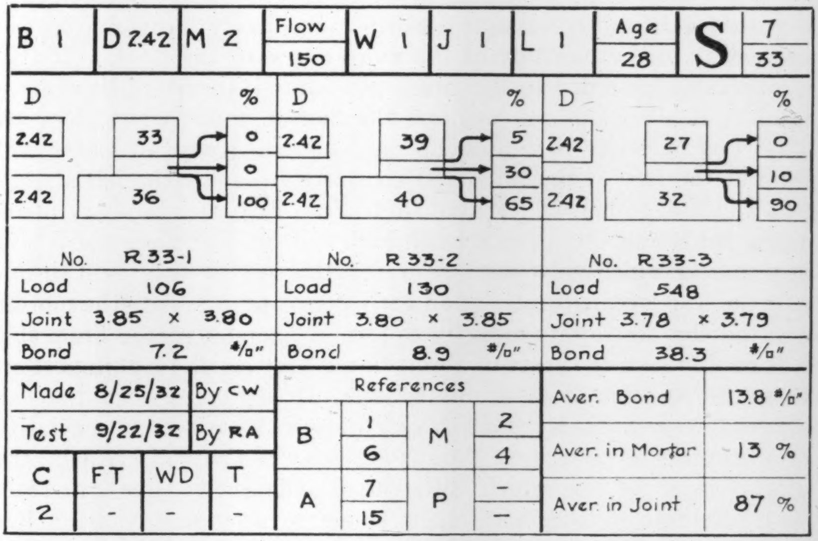


FIG. 6.—Tension Bond Test Specimen.

With these experiences in mind the variables considered in making up the test specimens were as follows (exclusive of brick type and mortar mix):

1. Specific-mass gravity of brick (laboratory key D).
2. Percentage of total absorbed water in brick (laboratory key W).
3. Thickness of joint (laboratory key J).
4. Load in joint (laboratory key L).
5. Method of curing (laboratory key C).
6. Method of treatment (laboratory key T).
7. Type of test specimen.
8. Age of specimen.

The brick used for any individual series must be selected to be sure that they have the same specific-mass gravity, and it is of course important that they be of the same manufacture. Specimens were laid up dry (W1), with 50 per cent of their total absorbed water (W2), and immersed for 2 hours and surface dried (W3). Three joint thicknesses have been used, namely, $\frac{1}{4}$ in. (J1), $\frac{3}{8}$ in. (J2), and $\frac{1}{2}$ in. (J3). Specimens were unloaded (L1), loaded with 100 lb. per unit (L2), and with 200 lb. per unit (L3). Specimens were cured by both methods (C1 and C2) as before described. In determining the type of test specimens, many trials were made and the specimen shown in Fig. 6 is now used exclusively. These tests are giving us very uniform tension breaks which may easily be compared with the tensile strength of the mortar. Specimens have been tested at 28 days, 3 months, 6 months, and 1 year, the storage of specimens in most of these cases being out-of-doors after having been cured for 28 days.



|  |  |  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|
| B 1 | D 2.42 | M 2 | Flow<br>150 | W 1 | J 1 | L 1 | Age<br>28 | S | 7<br>33 |

|  |  |  |
|---|---|---|
| D %<br>2.42 33 0<br>0<br>2.42 36 100 | D %<br>2.42 39 5<br>30<br>2.42 40 65 | D %<br>2.42 27 0<br>10<br>2.42 32 90 |
| No. R33-1 | No. R33-2 | No. R33-3 |
| Load 106 | Load 130 | Load 548 |
| Joint 3.85 × 3.80 | Joint 3.80 × 3.85 | Joint 3.78 × 3.79 |
| Bond 7.2 #/□" | Bond 8.9 #/□" | Bond 38.3 #/□" |

|  |  |  |  |
|---|---|---|---|
| Made 8/25/32 | | By CW | |
| Test 9/22/32 | | By RA | |
| C | FT | WD | T |
| 2 | - | - | - |

| References |  |  |  |
|---|---|---|---|
| B | 1<br>6 | M | 2<br>4 |
| A | 7<br>15 | P | –<br>– |

|  |  |
|---|---|
| Aver. Bond | 13.8 #/□" |
| Aver. in Mortar | 13 % |
| Aver. in Joint | 87 % |

FIG. 7.—Assemblage Record.[1]

Records for these tests are kept on cards such as shown in Fig. 7. It will be noted that careful data are recorded for each brick, for bond strength, for percentage of break at the brick and in the mortar, and that three specimens are used for each type of test where the variables are cross-referenced to the variables mentioned above. The position has been taken that a brick and mortar assemblage which will fail 50 per cent or more in the mortar is far superior to one which will not, regardless of the actual strength in pounds per square inch. Our reason for this is based upon the fact that it is more important to insure a "bond layer" at the brick line, than it is to insure no break in the mortar. A large number of such comparative tests have been conducted and space does not permit of their inclusion here.

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[1] This card is presented as an example of record only, although the data are actual results of tests.

## REACTIONS WHICH PRODUCE BOND

It is extremely important that the brick and mortar start out with intimate contact. This is necessary in order to allow the deposition of the "bond layer" which immediately forms due to the movement of the moisture of the mortar toward the brick. If this movement is retarded or too much accelerated, failure of bond may result. If a brick is too wet and the mortar is one which depends upon loss of water to consolidate and harden, a film of water will form at the face of the brick and completely destroy the initial contact and lead to high differential shrinkages, leaving a capillary plane at the brick line. If a brick has a high and rapid suction and is laid in a mortar which must not lose water, this loss of water will so impair the water-cement ratio at an early age as to granulate the mortar and thereby destroy its capacity to form a "bond layer." From this it is readily seen that rate of absorption is very much more important than the amount of total absorption. It is further seen that an attempt to wet brick before laying may so impair their regularity of absorption as to destroy the homogeneity of the wall. Again, the mortar must be one which will remain sufficiently "live" to react with alternate wetting and drying so as to carry on a more or less continuous building up of the "bond layer." This, if true, would seem to exclude extremely dense bricks and mortars which complete their hydration in a very short time, and which in turn obstruct the movement of water to too great a degree.

Furthermore, a mortar which is not so constituted as to allow dissolution and recrystallization at the plane of the brick will be unable to build up and heal slight ruptures produced by differential shrinkages. Again, a mortar so constituted as to produce a crystalline matrix at the brick line will have the same movement as the brick. This matrix will gradually seal the mortar at the brick line by withdrawing those compounds from the interior of the mortar which aid in the formation of this "bond layer." If a brick is so constituted as to approach in its ratio of expansion or contraction that of the substances which may be formed at its surface, it will aid in producing a bond. If, on the other hand, this is not possible, the brick should not be used for exterior walls.

All of this discussion is predicated upon the hypothesis that the movement of water in and through the mortar and brick produces a layer at the brick surface, and that such movement of water is not only desirable but necessary. We will consider this hypothesis briefly by a study of thin sections made for study using the petrographic microscope from actual specimens which have shown known tendencies. Again let us confine our considerations to a few outstanding examples which illustrate a general tendency. We will consider again mortars M2, M4 and M5 with brick types B3, and B5. Photomicrographs of these are shown in Figs. 8 to 12.

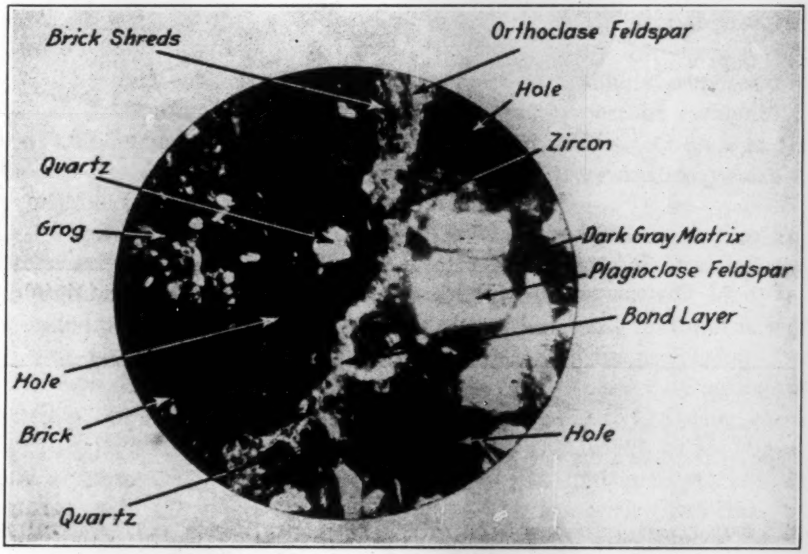

FIG. 8.—Photomicrograph of Brick and Mortar Interface (B3–M2) (× 46).

Figure 8 (B3–M2) shows the most continuous "bond layer" seen for this mortar. The width is slightly variable, but at all times there is a layer adjacent to the brick. There are numerous fragments of brick, from well-defined to indefinitely outlined particles particularly noticeable in the "bond layer." These fragments may be of two kinds. They may, first, be fragments of the original brick now entirely separated from the main mass of the brick, or they may, second, be projections still connected with the brick, but due to the orientation of this section they appear to be separated. In either

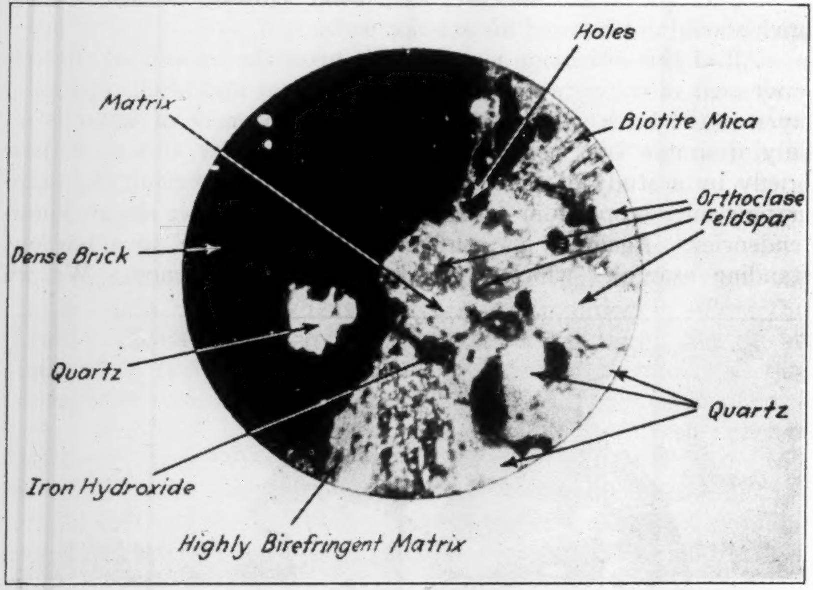
Holes
Matrix
Biotite Mica
Orthoclase
Feldspar
Dense Brick
Quartz
Quartz
Iron Hydroxide
Highly Birefringent Matrix

FIG. 9.—Photomicrograph of Brick and Mortar Interface (B3–M4) (× 46).

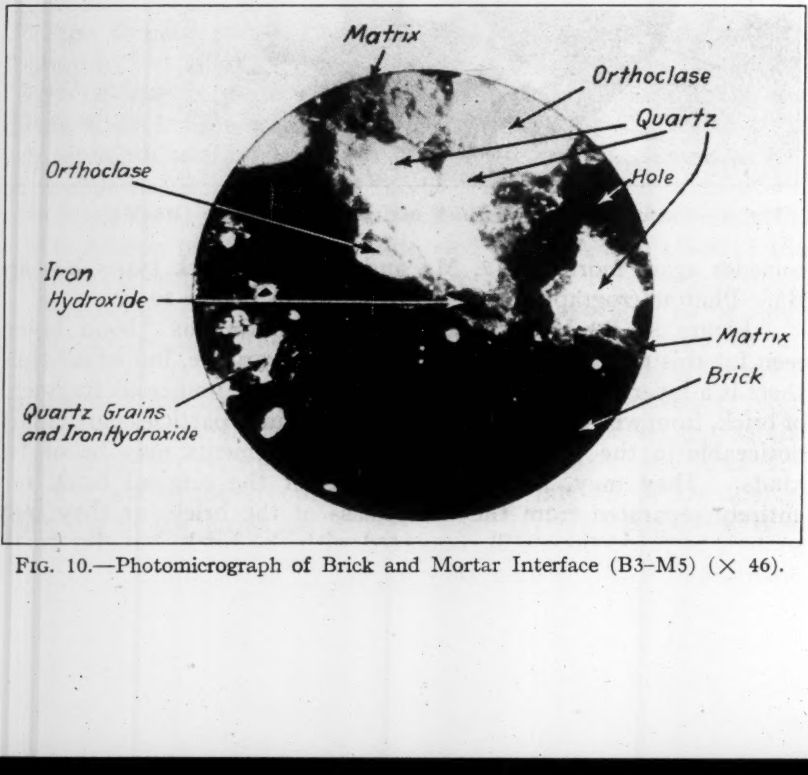
Matrix
Orthoclase
Quartz
Orthoclase
Hole
Iron
Hydroxide
Matrix
Brick
Quartz Grains
and Iron Hydroxide

FIG. 10.—Photomicrograph of Brick and Mortar Interface (B3–M5) (× 46).

case, some of these fragments show wispy indefinite outlines, that is, the outline does not seem to be as it was originally. The only probable reason for this seems to be attributable to a process whereby some of the brick of the fragment has been absorbed and replaced by the material now in the "bond layer."

The particularly good contact between the mortar and the irregular surfaces of the brick is due in part to the ideal physical relationships between the mortar and the brick in this particular specimen, but this does not necessarily preclude some replacement and infiltration of the brick by the mortar. The actual physical tearing off and forcing aside of brick fragments by pressure exerted by a growing crystal may or may not be true; it probably is not true.

The dark gray material of the matrix is probably composed of one or a combination of several of the more important elements of portland cement. The material of this matrix in this section is in the main isotropic but contains sparsely scattered, very minute grains of highly birefringent material.

Figure 9 (B3–M4) shows the structure of the mortar joint for this brick and mortar. The highly birefringent material is developed throughout the whole width of the mortar section as seen here; no rounded or any other shaped patches of the dark gray material is seen in this section. Careful examination under high magnification does not show any variation in character even up to the very edge of the brick. The contact is not smooth and flowing nor, on the other hand, is it so jagged and indented (indented into the brick) as in Fig. 8; nor are there so many fragments of the brick in the mortar. The mortar is in all places in very intimate contact with the brick (no line of separation is seen). "Infiltration," as we understand it, is not well developed in this section.

Figure 10 (B3–M5) shows a section which, under non-polarized light is yellowish and cloudy as compared with the white, clear material of the mortar M4. The birefringence of this mortar (M5) is also slightly lower than that of the mortar M4 but nothing comparable with the dark gray material of mortar M2. The "bond-layer" material is developed throughout the width of the section. There is a very indefinite cloudiness in places, but the material does not approach the darkness of the dark gray material seen in Fig. 8. It is to be noted that around the edges of some of the sand grains there is developed some highly birefringent material. This section is slightly thicker along one edge and the matrix seems to be the same as that shown in Fig. 9. The contact is very irregular and there are many

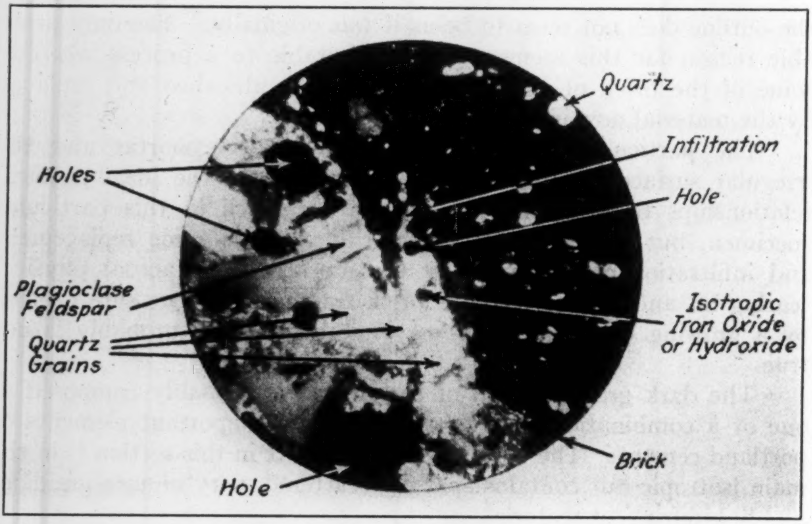
Quartz
Infiltration
Holes
Hole
Plagioclase Feldspar
Isotropic Iron Oxide or Hydroxide
Quartz Grains
Brick
Hole

FIG. 11.—Photomicrograph of Brick and Mortar Interface (B5–M5) (× 46).

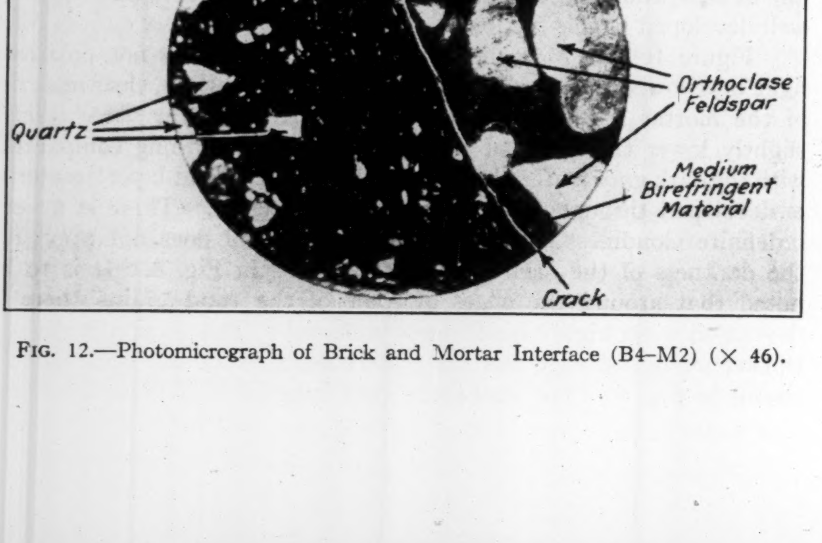
Brick
Crack
Quartz
Orthoclase Feldspar
Quartz
Medium Birefringent Material
Crack

FIG. 12.—Photomicrograph of Brick and Mortar Interface (B4–M2) (× 46).

curving projections of the brick into the mortar. These are more evident here than in Fig. 9. This would seem to indicate that this mortar has greater powers of infiltration, if the irregularities can be assigned to infiltration.

Figure 11 (B5–M5) shows no apparent break between the brick and the mortar; excellent infiltration is a common characteristic; and the contact is good. The matrix of the mortar shown in this field is all of the same order of birefringence and no banding is shown next to the brick. No low order birefringent material is shown near the brick or elsewhere in the section. This indicates that no portland cement elements of low-order of birefringence are present. The infiltration is complete, deep and sinuous.

Figure 12 (B4–M2) is an illustration of a very common phenomenon where mortars with high portland cement content are used. This crack, which is discontinuous, but general, is approximately 0.0004 in. in width. It is also apparent that the "bond layer" has not formed at the crack and an examination under higher magnification shows that no "bond layer" is present immediately at the brick surface.

A mortar having properties which lead to the formation of a "bond layer" may well be expected to induce healing within itself and to experience crystal growth within its voids, thus becoming much denser itself. This property, autogenous healing, has been known to exist in concretes, but for entirely different reasons. A mortar high in its lime content will have a sufficient supply of calcium hydroxide in its mass to provide a generous source for further chemical action. It is also very probable that an irregular break in the mortar layer itself will lead to much more rapid healing than the phenomenon exhibited in the early stages at the plane of the brick. It is further worthy of note that the thicker a joint the weaker the tensile bond, particularly where mortars containing large proportions of lime are involved. This may be due to the removal of water and its dissolved contents from the central plane of the mortar leaving it not only with greater voids but less prepared to harden or set.

Differential shrinkages set up in a brick and mortar assemblage which is more or less devoid of a "bond layer" as described and in which the mortar is such as to rapidly obtain its final hardness will be conducive to separation at the plane of the brick. On the other hand, such shrinkages set up in a mortar which has developed a "bond layer" at the plane of the brick and which possesses enabling characteristics to allow continuance of such bond-producing action will, if it breaks at all, separate in the mortar itself. This break will subsequently build up by crystal growth in exactly the same fashion as the initial layer and take care of such movements in this way so as to remove eventually sufficient of the differential shrinkage to allow the shear strength of the mortar to carry the developed strain.

## AN HYPOTHESIS

Before proposing any hypothetical conditions it may be well to call attention to some of the outstanding indications (not conclusions) which have been shown to be justified by more than two years of investigation on the part of the author:

1. Brick and mortar characteristics considered separately will yield no criterion for permeable bond.
2. All brick will not react favorably with all mortars, but must be studied to find their proper complementary mortar.
3. Uniformity of brick structure is indispensable in selecting a mortar for use with the brick.
4. It is absolutely essential that a brick possess from 5 to 10 per cent total absorption, by immersion for 48 hr., and that its rate after the first 5 to 10 min. be uniform and gradual for several hours.
5. Absorption tests are far more reliable in deciding the desirability of a brick for external use than any strength tests now used.
6. Mechanical bonds produced by wire-cutting, frogs, etc., do not materially affect the tensile bond strength, nor the final bond layer.
7. Excessive density (weight per unit volume) in a mortar is just as objectionable as it is in a brick.
8. Cement and lime are equally important constituents of a good mortar. Neither may be omitted where all the usual conditions of exposure and construction exist.
9. Highly absorbent brick require mortars with high lime content, whereas brick with reasonable lower percentages of absorption (about 5 per cent) may well have the lime content reduced to not less than equal parts of lime and cement by volume.
10. Extremely dense brick or brick which have high differential shrinkages with respect to the mortars used and which present a difficult problem, must be laid in high lime content mortars to afford as much protection and plastic contact as possible, going perhaps to a straight, lime-sand mortar.
11. Loaded walls are more watertight than curtain walls. Intimate contact is thus advantageous.
12. Thin joints are more effective than thick joints.

The following hypothesis is offered for consideration:

> A leakless brick masonry wall may be produced by an assemblage of brick and mortar where, first, the relation of the absorptive power of the brick to the amount of water in the mortar is such that the initial movement toward the brick plane sets in early and continues at an even rate for several hours; and where, second, the mortar is so constituted as to permit such loss of water and still provide a soluble product which in the process is concentrated at the brick plane and is available thereafter for a long period of time to form a compound with a minimum of differential shrinkage with the brick, the assemblage being capable by its porous structure to permit repetition of such action.

The composition of the "bond layer" presents many difficult petrographic and crystallographic problems. Study so far directed toward this end seems to indicate that this layer, regardless of the mortar mix, is a compound which we may call "X." The possibilities are that this compound "X" contains calcium; and furthermore, the likelihood of this calcium being in the form of a hydroxyl or carbonate is stronger than that of its being a silicate. Further study is being conducted to determine definitely, if possible, the exact composition of the above-mentioned compound "X."

## DISCUSSION

Mr. J. W. McBurney.[1]—With reference to Mr. Voss's paper, I must take exception to several of the "indications" given in the final summary. Indication 4 states: "It is absolutely essential that a brick possess from 5 to 10 per cent total absorption, by immersion for 48 hr., and that its rate after the first 5 to 10 min. be uniform and gradual for several hours." If this is correct, no dry walls have ever been laid in New York City, Los Angeles, or several other communities in the United States, because the absorption of brick of those communities is above 10 per cent by 48-hr. cold submersion. May I point out that the four brick which are described in this paper gave 48-hr. cold absorption of from 3.3 per cent up to 9.6 per cent. Over 50 per cent of the brick product of the United States is outside of these limits. I do not believe that the four brick used represent a fair cross-section of the brick production of the United States.

Indication 6 reads: "Mechanical bonds produced by wire-cutting, frogs, etc., do not materially affect the tensile bond strength, nor the final bond layer." I cannot agree with that at all. I believe in Mr. Withey's paper[2] he found considerably improved bond strength for the wire-cut brick, and I can cite quite a few determinations of other investigators to the effect that higher bond strengths are produced by roughening the flat of the brick.

Are not indications 9 and 10 somewhat contradictory? Reference is made to the impossibility of getting bond between a glass brick and a cement mortar. For some time past I have had under observation two rectangular glass specimens which have been cemented together with a very rich cement mortar, about 1:2, and the bond is holding very well.

Mr. W. C. Voss.[3]—One of the objections that Mr. McBurney has raised is the question of the representative character of the brick examined. It was not intended that they be representative, and I have therefore not drawn conclusions.

As far as the justification of indications 9 and 10 is concerned, the first indication is included for the purpose of showing that in order to get any kind of a bond with a slowly absorbent brick, or a low-absorbent brick, we must have plasticity in the mortar, and the only way we can get that is by adding a soluble plasticizer of some kind. Further, that if there is any absorption in the brick there will be a lot of this soluble product able to pass to the plane of the brick, producing a product as close as possible in its volume change to the brick itself. I know that these two indications sound contradictory. I put the last one in because many people want to lay brick that are extremely dense, and the only mortar which in general has given me over 50 per cent break in the mortar using these dense brick was a 1:3 straight lime-sand mortar.

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[1] Research Associate of the Common Brick Manufacturers' Assn. at the U. S. Bureau of Standards, Washington, D. C.

[2] M. O. Withey, "Tests on Brick Masonry Beams," see p. 651.

[3] Consultant in Architectural Construction and Professor of Building Construction, Massachusetts Institute of Technology, Cambridge, Mass.

When I say "bond strength," I do not mean pounds per square inch only. I mean percentage of break at the brick plane also, and this has been more than 60 per cent where the mortar and brick combination was such as to produce a failure because of lack of bond there. Specimens with frogs and scored surfaces have broken just as completely at the plane of the brick as did those with smooth faces.

I have not been able to conduct a series of tests that have enabled me to cause volume changes to take place over long periods of time, but I think it is a matter of common information that the walls that are causing this trouble today are the ones where relatively dense brick have been laid with relatively strong and dense mortar. To use 28-day results is dangerous.

I do not think that I have found the answer by any means; I do not think anybody else has, because in a good many instances tests have been conducted on a group of brick about which the investigator knew little or nothing; simply because they came from the same place and the same kiln he thought they were alike. I have plenty of evidence to show that six brick selected from different places in the same kiln, under the same temperature, showed variations in compressive strength from 2000 to 7000 lb. per sq. in., and in absorption from 2 up to about 12 per cent. It is the processing of the brick which must be studied, and I think that the brick manufacturers can very well afford to develop a processing so as to get a capillary structure that will produce the correct result.

MR. F. O. ANDEREGG.[1]—Mr. Voss has developed an ingenious hypothesis for the development of a "bond-layer" and its maintenance by dissolution of lime compounds which are under stress, with redeposit in such a way as to heal up the weakened spot. The latter effect is often spoken of as the "flexibility" of the mortar, and in this action the compounds of lime are apparently playing a principal rôle. This has been observed wherever lime has been used in masonry and to a less extent in portland-cement concrete; it is also characteristic of natural cements. It is important also where other movements, such as vibration or windsway, occur, to provide adequate flexibility in the mortar.

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[1] Consulting Specialist on Building Materials, Pittsburgh, Pa.

While lime is a mighty fine plasticizing material, other plasticizing materials are available, such as ground shale. I know of many instances where 15, 20 and 25 per cent ground shale has been used with portland cement to give very fine results. I know of cases where such mortar has withstood 200 cycles of freezing and thawing. The bond between the brick and shale seems to be very good. It is, however, in my opinion a little deficient in elasticity, in ability to give a little as the volume changes. To obtain this, lime is one of the most important constituents we have, but lime changes to carbonate, and the limestone itself may be incorporated in the mortar giving the same sort of flexibility as lime.

Considerable emphasis has been laid on the absorption characteristics of the unit and the range indicated does yield best results, but it should be pointed out that with proper attention to mortar design, with adequate specifications for, and supervision of workmanship, combined with suitable design of the wall and its auxiliary construction, it is readily possible to secure quite satisfactory practical construction with units having very low initial and total absorption, as witness the house built out of glass units at the Chicago Century of Progress.

Because of the sliding and slumping tendency of low-absorption units laid with high-lime mortars and because of the low rate of hardening of such mortars, especially in cooler weather, the use of this combination becomes hardy practical, if not actually dangerous.

If the brick themselves are permeable, it is impossible to build a watertight wall by paying attention only to the "bond layer." In such a case, provision must be made to take care of any moisture getting into the wall; and, indeed, this is the only safe and satisfactory method of building a watertight wall where appreciable exposure to driving rains occurs.

The importance of intimate initial contact between brick and mortar, as essential to the development of bond, cannot be too highly emphasized. To secure this contact, thin joints and pressure are necessary. If masonry units could be manufactured with sufficient uniformity of dimension, a joint thickness ranging from $\frac{3}{16}$ to $\frac{1}{4}$ in. would be ideal, and the use of any joint thickness greater than $\frac{3}{8}$ in., especially in cold weather, is inviting leakage.

I know from personal experience with the best brick masons I could secure, working as carefully as possible, it is seemingly impossible to get a perfect contact all the way between the brick and the mortar. That does not matter in a great many walls. The only solution is to provide within the walls, within its design, a means of draining off the water and ventilation for carrying it off. If this is done you will have very satisfactory walls, and you do not need to be so extremely fussy about some of the brick and mortar.

MR. R. E. DAVIS.[1]—One very important thing that has been partially brought out, but upon which I should like to place additional emphasis, is the necessity for having a mortar with a relatively low volume change. We should not go to very rich mortars, because almost without exception mortars which are rich either in lime or in cement, in spite of the high bond strength that may be obtained (unless subjected to alternations of wetting and drying, or freezing and thawing), will produce large volume changes. There is an intermediate ground between the rich mortars and the lean mortars where best results will be obtained.

We must have, first, a mortar which is not porous, which is not absorbent, which possesses strength; and, second, a mortar with minimum volume change.

MR. J. R. DWYER.[2]—What is the criterion, if any, that governs the selection of a particular cement?

MR. VOSS.—The criteria used involved an examination of the proportion of tricalcium silicate, and this was slightly higher than the general run of ordinary cement. I think it has been established that the greater the proportion of tricalcium silicate the less we have of volume changes later on; and the greater the proportion of dicalcium silicate the larger the volume change in producing it.

Replying to some of the comments made, the paper was not intended to deal with workmanship. If a mortar has the characteristic which enables it to build a bond layer at the plane of the brick and thereby causes a failure, if any, in the mortar, it probably has the characteristic to heal that break later on.

In conclusion let me say that I do not profess to have more than scratched the surface. I do not profess to have described all the brick characteristics of the United States, or all of the mortars. But I do think that we will have a story as far as the scientific relation of the porous structure of the brick with the action of a mortar goes.

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[1] Professor of Civil Engineering, In Charge of Engineering Materials Laboratory, University of California, Berkeley, Calif.

[2] Research Associate, U. S. Bureau of Standards, Washington, D. C.

# FACTORS IN THE PRESENTATION AND COMPARISON OF PARTICLE SIZE DATA

By E. J. Dunn, Jr.[1] and John Shaw[2]

## Synopsis

This paper discusses certain important factors affecting the presentation and comparison of particle size data. Some problems of measurement are discussed briefly. Various graphical forms of presenting data are listed and summarized. Four accurately sized products giving diversified ranges of size are used in certain illustrative graphs. The present status and use of representative functions of particle size data is described. Need for standardization of methods of particle size analysis and presentation of data is emphasized.

## Introduction

During the past few years, with the realization of the rôle played by fineness in scientific and industrial fields, the need for adequate presentation of fineness data has become more and more apparent. Due to the diversity of such research, investigators in the field of particle size have developed various techniques of sizing, each with its individual manner of presenting data. Many developments have been made to fit the needs of the particular user and to portray his ideas rather than to bring out any basic concept of distribution. This makes very difficult the comparison of fineness data from different sources.

Broadly speaking, the purpose of particle size measurement is to discover the true frequency distribution of particle size. The observed data for any representative sample, obtained by microscopic count of particle size, consists in a frequency distribution which gives the frequencies of occurrence for specific ranges of particle size. The observed frequency distribution serves as the basic data from which may be derived (*a*) certain representative constants or (*b*) modified relative frequency distributions obtained by transformation, such as per cent by weight (per cent of total weight of the sample contributed by particles of each class size) *versus* size, which often contain the essential information or are more convenient to use than the raw data in interpretation of results.

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[1] Research Laboratories, National Lead Co., New York City.

[2] Department of Chemical Engineering, Columbia University, New York City.

## PROBLEMS OF MEASUREMENT AFFECTING PARTICLE SIZE DATA

The extent to which particle size data may be used depends on the accuracy with which such data are obtained. It appears to the authors that distribution curves presented for materials are sometimes based on too few data without adequate consideration to the errors of the particular technique employed, such as could be disclosed by repeated sets of measurements under the same essential conditions. Where the true distribution of a material is sought, it is important to discover how much of the observed irregularity in a distribution is due to errors in technique and sampling and how much is due to the material itself.

Since most methods of particle size analysis are calibrated microscopically, it is particularly fitting to discuss some problems of microscopic measurement which affect the accuracy of the data. Sampling and mounting of the material are extremely important in microscopic measurement because of the minute amount used for test. The technique used in handling and mounting may cause segregation of certain sizes. The sample for analysis should be well mixed to insure uniformity. Portions of the uniform samples mounted several different ways and in several media should then be studied visually under the microscope. This visual study suggests the type of mount to use. After the mounts are prepared for counting they should be checked to see if they qualitatively conform to the findings in the preliminary examination as to the ratio of sizes present. The visual study probably is the most satisfactory check on the sampling and mounting. To guard against systematic errors, it is suggested that the analysis be duplicated by an entirely different technique of mounting for any material having a wide range of particle size.

Inadequate designation of the "diameter" used in measurements limits the use or comparison of data. Investigators in various lines use the horizontal, maximum, minimum, average of two visible, or harmonic mean of the three diameters, and some apply shape factor corrections to express their results. For relative comparison between samples of any one material, any diameter gives to some extent an indication of the size relations. However, for the comparison of different materials, that diameter which is the nearest representation of the actual physical character of the particle should be used. If surface or volume are to be considered, the diameter used should be one that gives the closest possible approximation to the actual surface or volume. For many materials this may necessitate a correction factor due to the deviation of the third dimension of the particles from the two dimensions visible in the microscope. Whatever diameter is taken should be clearly designated, and the explanation as to how it is obtained and used brought forth in the presentation of the data.

Inaccuracies are also present in the actual measurement of the particles. Certain assumptions are necessary in all types of particle size measurement. In general, no linear measurement approaches the exact relation of surface or volume of the particles. The application of shape factors is only a single step in the endeavor to bring the results nearer to the theoretical. The method used for applying shape factors is an important variable. In addition, the various techniques used in sedimentation, elutriation, and sieving affect the value of diameter recorded and should be considered in the comparison of results where such diameter is measured indirectly and not calibrated microscopically. All of these factors have a definite contributory effect on the distribution curve determined for the material, and should be discussed in detail if the results presented are to be of value to others.

Often the number of particles counted for an analysis is so small as not to be truly representative of the material. The number of particles which must be counted depends entirely upon the range of sizes and their respective frequencies. In the authors' experience a material with a size range of approximately 8 microns down containing a large percentage of fines necessitates a count of at least several thousand particles or the equivalent through "corrective counts" to yield satisfactory results. Considerably less counting is required for coarser materials or fractionated portions. In any case the number counted should be sufficiently great so that additional counting does not alter the shape of the distribution curve significantly. From the standpoint of discovering causes of variability in observed results, less information can be obtained by making one set of measurements on a very large number of particles than by making several rational sub-sets of measurements, each sub-set representing a proportionately smaller number of particles.

Fundamentally the measurement of each particle for size would give the best distribution. Practically what is done in microscopic work is to classify particles into a series of predetermined intervals of size. Grouping into intervals in itself introduces errors which may be serious if the number of intervals is too small. The choice of the number of intervals and of the interval boundaries depends of course on the use to be made of the data. If the object is to determine the frequency distribution of size of a material or to derive therefrom one or more of the representative constants, the number of intervals should ordinarily be not less than 10. Too few intervals may alter materially the apparent shape of the distribution as well as the constants obtained from the data. On the other hand, a large number of intervals, say over 20, greatly increases the labor in making a determination, and does not necessarily increase the value of the data correspondingly, from the standpoints of accuracy and interpretation.

TABLE I.—GRAPHICAL FORMS.

| ORDINATE | SCALE | ABSCISSA | SCALE |
|---|---|---|---|
| No. 1.—Observed frequency by count or per cent | Linear | Size in microns | Linear |
| | Logarithmic | Size in microns | Linear |
| | Logarithmic | Size in microns | Logarithmic |
| No. 2.—Computed per cent by weight | Linear | Size in microns | Logarithmic |
| | Linear | Size in microns | Linear |
| No. 3.—Computed per cent by surface | Linear | Size in microns | Linear |
| | Linear | Size in microns | Logarithmic |
| No. 4.—Computed per cent per micron by frequency surface or weight | Linear | Class interval | Linear |
| | Linear | Average diameter of interval | Linear |
| No. 5.—Cumulative per cent by frequency surface or weight less or greater than stated size | Linear | Size in microns | Logarithmic |
| | Linear | Size in microns | Linear |
| | Probability | Size in microns | Logarithmic |
| | Probability | Size in microns | Linear |
| No. 6.—Cumulative per cent by weight | Linear | Reciprocal of diameter | Logarithmic |
| No. 7.—Per cent per micron per gram | Linear | Surface mean diameter | Logarithmic |

Due to the practical limitations imposed, equal intervals are limited in use to small ranges of particle size. For wide ranges of size, it is usually necessary to increase the interval width with increase in particle size. Intervals selected according to a fixed geometric ratio (each interval equals preceding interval times a constant) prove very satisfactory for general use. Such intervals appear equal in width on a graphical chart which uses a logarithmic scale for size. Intervals chosen in this way require decimals to express the boundaries of the intervals—a disadvantage that can often be overcome without great error by using approximate values.

## GRAPHICAL PRESENTATION OF DATA

The observed data obtained by microscopic count of particle size consists of a frequency distribution, giving the frequencies of occurrence for specific ranges of particle size. The presentation of this observed distribution in tabular form is not readily interpreted nor is it, in general, adequate for the practical use made of such data. A presentation graphically of the observed distribution or of certain transformed distributions is often helpful in the interpretation and use of the data.

Graphs used to present data should be, if possible, drawn up on a basis which does not require reference to the data for explanation. Graphical distributions of various types have been developed and used by different investigators to assist in a study of irregularities in the shape of distributions, to aid in discovering non-homogeneity or causes thereof, and to make quick comparisons of distributions for different materials. In general, it is customary to use particle size or diameter for the abscissa scale and observed or cumulative frequency for the ordinate scale. The ordinate scale may be some function of the frequency observed by microscopic count, such as surface or weight. Different investigators use various combinations of linear, logarithmic, and probability scales for the coordinates of the graph. Some idea of the variety of combinations that have been observed in the literature is given in Table I.

When it is desired to portray graphically the shape of a distribution, it should be plotted on a basic unit interval. Three types of graphs that do this are:

1. Per cent per micron *versus* size on a linear-linear scale.
2. Per cent *versus* size on a linear-linear scale when equal intervals of measurement are selected.
3. Per cent on linear scale *versus* size on logarithmic scale when intervals of measurement are selected according to a fixed geometric ratio.

If the intervals of measurement are not equal or are not based on a geometric ratio, graphs of types 2 and 3 can be prepared only after an adjustment has been made to give the interval equality which these types of graphs demand. Type 1 imposes no such restrictions on the intervals of measurement.

If the purpose of the graphs is to show cumulative percentage above or below the various sizes, a probability scale appears to be best for the ordinate, with optional use of linear or a logarithmic abscissa scale. For this type of graph it makes no difference what intervals of size are used for measurement. Data from different

### TABLE II.—FINENESS DATA FOR SILICA SAND (NO. 3).

| Interval Boundaries, microns | Average Diameter, microns | Observed[a] Frequency | Per cent Frequency | Per cent Weight | Cumulative per cent by Frequency Undersize | Per cent per Micron by Weight |
|---|---|---|---|---|---|---|
| 104 to 124 | 114 | 0.00186 | 0.000000031 | 0.18 | 100.0000 | 0.009 |
| 88 to 104 | 96 | 0.001128 | 0.0000000188 | 0.07 | 100.0000 | 0.004 |
| 74 to 88 | 81 | 0.00192 | 0.000000032 | 0.07 | 100.0000 | 0.004 |
| 55.5 to 74 | 64.75 | ........ | ....... | 0.00 | 100.0000 | 0.000 |
| 37 to 55.5 | 46.25 | 5.4 | 0.00009 | 3.33 | 100.0000 | 0.180 |
| 27.75 to 37 | 32.38 | 36.6 | 0.00061 | 7.98 | 99.9999 | 0.862 |
| 18.5 to 27.75 | 23.13 | 205.2 | 0.00342 | 15.86 | 99.9993 | 1.714 |
| 13.88 to 18.5 | 16.19 | 522 | 0.00870 | 14.25 | 99.9959 | 3.08 |
| 9.25 to 13.88 | 11.56 | 1 152 | 0.01920 | 11.13 | 99.9872 | 2.41 |
| 6.94 to 9.25 | 8.09 | 2 274 | 0.03790 | 7.72 | 99.9680 | 3.34 |
| 4.63 to 6.94 | 5.78 | 6 084 | 0.10140 | 7.34 | 99.9301 | 3.17 |
| 3.47 to 4.63 | 4.05 | 14 634 | 0.24390 | 6.25 | 99.8287 | 5.41 |
| 2.313 to 3.47 | 2.89 | 47 328 | 0.78880 | 7.13 | 99.5848 | 6.17 |
| 1.734 to 2.313 | 2.02 | 110 880 | 1.84800 | 5.89 | 98.7960 | 10.18 |
| 1.156 to 1.734 | 1.44 | 240 680 | 5.67800 | 6.40 | 96.9480 | 11.06 |
| 0.578 to 1.156 | 0.87 | 1 333 800 | 22.23000 | 5.43 | 91.2700 | 9.40 |
| 0.000 to 0.578 | 0.289 | 4 142 400 | 69.04000 | 0.97 | 69.0400 | 1.68 |

[a] Determined data from which all other values are computed. The total number of particles counted for this sample is 22,550.

### TABLE III.—FINENESS DATA FOR BASIC LEAD CHROMATE (NO. 4).

| Interval Boundaries, microns | Average Diameter, microns | Observed[a] Frequency | Per cent Frequency | Per cent Weight | Cumulative per cent by Frequency Undersize | Per cent per Micron by Weight |
|---|---|---|---|---|---|---|
| 12 to 16 | 14 | 20 | 0.08 | 8.28 | 100.00 | 2.07 |
| 8 to 12 | 10 | 222 | 0.87 | 44 56 | 99.92 | 11.14 |
| 6 to 8 | 7 | 381 | 1.49 | 22.49 | 99.05 | 11.25 |
| 4 to 6 | 5 | 453 | 1.76 | 13.65 | 97.56 | 6.83 |
| 3 to 4 | 3.5 | 364 | 1.42 | 4.04 | 95.80 | 4.04 |
| 2 to 3 | 2.5 | 452 | 1.76 | 2.66 | 94.38 | 2.66 |
| 1.5 to 2 | 1.75 | 482 | 1.88 | 1.56 | 92.62 | 3.12 |
| 1.0 to 1.5 | 1.25 | 993 | 3.87 | 1.18 | 90.74 | 2.36 |
| 0.5 to 1.0 | 0.75 | 3 900 | 15.23 | 1.00 | 86.87 | 2.00 |
| 0.25 to 0.5 | 0.375 | 18 372 | 71.64 | 0.58 | 71.64 | 2.32 |

[a] Determined data from which all other values are computed. The actual number of particles counted for this sample is 26,104.

### TABLE IV.—FINENESS DATA FOR RED LEAD (NO. 1).

| Interval Boundaries, microns | Average Diameter, microns | Observed[a] Frequency | Per cent Frequency | Per cent Weight | Cumulative per cent by Frequency Undersize | Per cent per Micron by Weight |
|---|---|---|---|---|---|---|
| 48 to 64 | 56 | 1 | 0.0000 | 0.08 | 100.000 | 0.005 |
| 32 to 48 | 40 | 69 | 0.0002 | 2.02 | 100.000 | 0.127 |
| 24 to 32 | 28 | 148 | 0.0004 | 1.50 | 100.000 | 0.187 |
| 16 to 24 | 20 | 455 | 0.0013 | 1.67 | 99.999 | 0.22 |
| 12 to 16 | 14 | 4 605 | 0.0131 | 5.80 | 99.998 | 1.45 |
| 8 to 12 | 10 | 30 309 | 0 086 | 13.95 | 99.985 | 3.48 |
| 6 to 8 | 7 | 80 100 | 0.230 | 12.53 | 99.899 | 6.30 |
| 4 to 6 | 5 | 415 000 | 1.189 | 23.79 | 99.669 | 11.89 |
| 3 to 4 | 3.5 | 733 000 | 2.10 | 14.58 | 98.48 | 14.58 |
| 2 to 3 | 2.5 | 1 468 000 | 4.21 | 10.52 | 96.38 | 10.52 |
| 1.5 to 2 | 1.75 | 2 564 000 | 7.35 | 6.30 | 92.17 | 12.61 |
| 1.0 to 1.5 | 1.25 | 4 730 000 | 13.54 | 4.26 | 84.82 | 8.52 |
| 0.75 to 1 | 0.875 | 5 960 000 | 17.07 | 1.83 | 71.28 | 7.32 |
| 0.5 to 0.75 | 0.625 | 7 810 000 | 22.37 | 0.90 | 54.21 | 3.59 |
| 0.25 to 0.5 | 0.375 | 11 110 000 | 31.84 | 0.27 | 31.84 | 1.08 |

[a] Determined data from which all other values are computed. The actual number of particles counted for this sample is 86,660 (total from two methods).

sources are easily compared by this means. Since a Normal Law distribution appears as a straight line when cumulative frequencies are plotted on a probability scale, this type of graph is occasionally used to show qualitatively whether an observed distribution is approximately normal in form.

Most of the other graphs listed in Table I are used for specific purposes rather than to give any basic concept of distribution. The graphs listed as Nos. 6 and 7 in Table I obtain surface relation from the weight data, since the mathematical integration of the area under the curve form is a constant function of surface.

Particle size data of four accurately sized materials are used to illustrate some of these graphs. Red lead, basic lead chromate, silica sand, and litharge have been selected to bring out distributions of widely varying methods of processing and fineness. The red lead is calcined and then disintegrated for use as a battery product. The basic lead chromate is a precipitated material. The silica sand is dry-ground material from a laboratory jar mill run of 14 hr. The litharge is made by sublimation and is commonly referred to as fume product. These samples should not be considered as representative of all materials of their respective class. The data for these materials are listed in Tables II, III, IV and V.

TABLE V.—FINENESS DATA FOR LITHARGE (NO. 2).

| Interval Boundaries, microns | Average Diameter, microns | Observed[a] Frequency | Per cent Frequency | Per cent Weight | Cumulative per cent by Frequency Undersize | Per cent per Micron by Weight |
|---|---|---|---|---|---|---|
| 6 to 8 | 7 | 0.3 | 0.01 | 0.68 | 100.00 | 0.34 |
| 4 to 6 | 5 | 1.3 | 0.05 | 1.32 | 99.99 | 0.66 |
| 3 to 4 | 3.5 | 21.3 | 0.87 | 11.19 | 99.94 | 11.19 |
| 2 to 3 | 2.5 | 102.6 | 4.16 | 30.00 | 99.07 | 30.00 |
| 1.5 to 2 | 1.75 | 149.5 | 6.07 | 21.18 | 94.91 | 42.36 |
| 1.0 to 1.5 | 1.25 | 252 | 10.23 | 19.89 | 88.84 | 39.78 |
| 0.75 to 1.0 | 0.875 | 276 | 11.20 | 7.47 | 78.61 | 29.88 |
| 0.5 to 0.75 | 0.625 | 612 | 24.82 | 6.05 | 67.41 | 24.20 |
| 0.25 to 0.5 | 0.375 | 1050 | 42.59 | 2.22 | 42.59 | 8.88 |

[a] Determined data from which all other values are computed. The actual number of particles counted for this sample is 3,833.

In obtaining the data for this work, except for silica, the average of the two visible diameters was used as the size of the particle. In the case of litharge and basic lead chromate, correction factors were applied for the thinness of the particles down to $1\frac{1}{2}$ microns. Particles of both samples were rolled over endwise in a viscous medium under the microscope and the third dimension measured. The actual volume and surface were then calculated from the three dimensions of the particle and compared with the volume and surface of a sphere, the diameter of which was the average of the two visible under the microscope. The ratio of the surface calculated from the three dimensions to the surface of the sphere is the correction factor for the surface calculations. The computed surface for the spheres are then multiplied by the correction factor for the final figures. The volume or weight correction factor was obtained the same way.

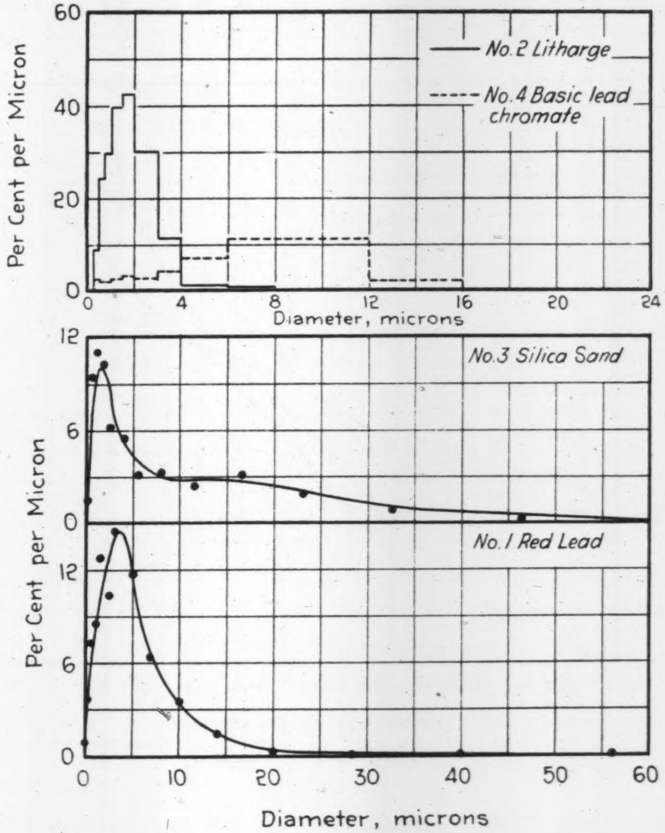

FIG. 1.—Relation of Percentage per Micron by Weight to Diameter.

In the particle size measurement of the silica sand the smallest visible diameter of the particle was measured for all material finer than No. 200. To join this material with the sieved portions, the smallest visible diameter was transformed to the sieve scale basis. This is obtained by getting the ratio of the diameter of particles just passing the sieve to the sieve aperture (see Figs. 1, 2 and 3).

## PRESENTATION OF DATA BY MEANS OF REPRESENTATIVE FUNCTIONS

The total information in a set of particle size measurements obtained by microscopic count, is contained in the observed frequency distribution. For certain uses to be made of published data, the entire observed distribution or one or more of the modified distributions, such as per cent by weight or per cent by surface *versus* size, may be needed to convey the essential information.

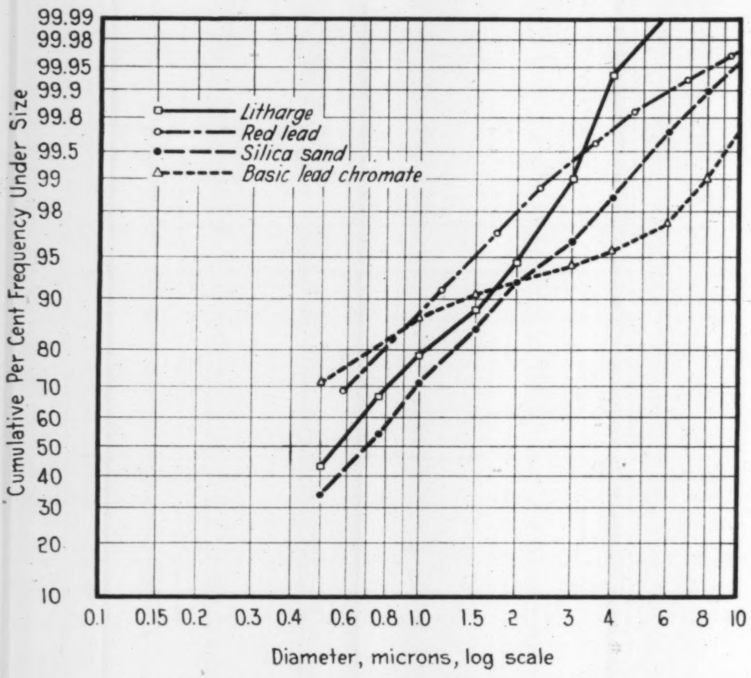

FIG. 2.—Cumulative Percentage Frequency Curves.

For many uses, however, such detail is not needed—the essential information is contained in one or more representative functions that may be derived from the observed frequency distribution.

These representative functions may be classed as:

(*a*) Averages or measures of central tendency, such as the arithmetic mean and the geometric mean.
(*b*) Measures of dispersion.
(*c*) Relative frequency or percentage above or below stated sizes.

Many of the representative functions used in particle size work are of the nature of averages that are computed from the observed and the modified frequency distributions. Among the several "average diameters" used are those based on length, surface, volume and weight, and the particle of average surface. From two of the more widely used "average diameters," namely, average diameter by surface mean and average diameter by volume, the specific surface and number of particles per unit weight may be computed.

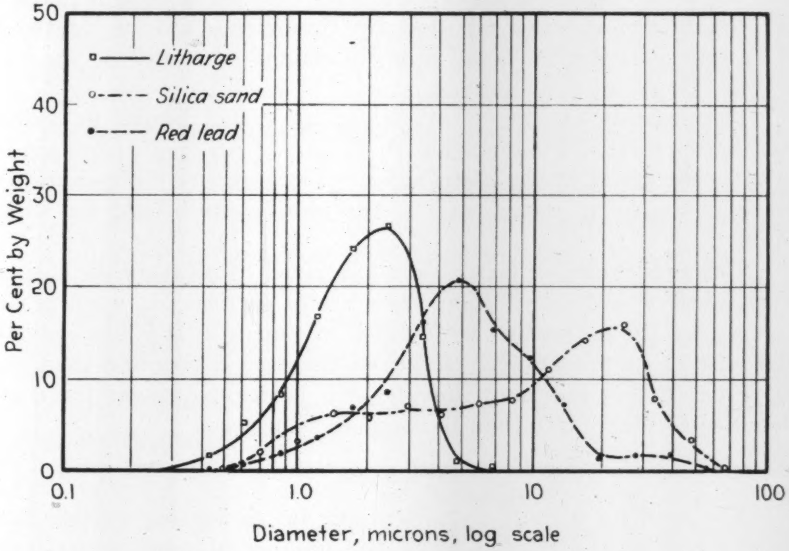

FIG. 3.—Relation of Per Cent by Weight to Diameter.

The points plotted in this figure for red lead and litharge do not correspond to the values given in Tables IV and V for the following reason: The boundaries of the intervals used in making these measurements do not progress precisely on a fixed geometric ratio. An adjustment was therefore made on the basis of equal logarithmic intervals giving equivalent values of per cent by weight which makes it possible to plot this type of graph.

Specific surface may also be computed from the particle of average surface if the total number of particles is known. Green[1] has published a summary of these constants.

The measures of dispersion that are usually used in particle size work are standard deviation, coefficient of variation, coefficient of uniformity and geometric standard deviation. No single function such as an average or a measure of dispersion, presented individually, is capable of giving much of the total information contained in a distribution. Much is contributed in regard to the average fineness and the spread by presenting both. When an average and standard deviation are presented, it should be stated for which distribution they apply. If geometric mean is given the logical accompanying measure of dispersion is the geometric standard deviation. Coefficient of variation is a percentage relation or ratio of standard deviation to the average (100 times standard deviation/average). The variability in standard deviation may be large while at the same time the percentage relation of variability may be small. A manual on presentation of data reported by Committee E-1 on Methods of Testing[1] describes in detail many functions that may be used to describe frequency distributions.

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[1] H. Green, "The Effect of Non-Uniformity and Particle Shape on Average Particle Size," *Journal*, Franklin Inst., Vol. 204, p. 713 (1927).

The relative frequency or percentage value above or below stated sizes such as the quartiles 25, 50 and 75-per-cent points aid in showing the trend of the distribution. Occasionally the percentage above or below a stated size is the only control used on some products.

Although skewness factors[1] are employed to measure the degree of asymmetry of a distribution, their use in particle size work is not believed by the authors to be justified in general. It is our opinion that when a more complete picture of the data is required than the above descriptive functions provide, it is better to present the complete distribution curve than to compute skewness.

## SUMMARY

The need for a more common basis for presenting particle size data is readily appreciated when the number of possible methods of presenting data are considered. Three factors of major importance in the collection and presentation of particle size data are:

1. Essential information regarding conditions under which data have been collected. Under this heading comes the conditions of sampling and mounting, expression of diameter, class interval selection and number of particles counted. Since there is such a wide diversity of results obtained by the various methods of analysis, all results should be reported according to the technique used. The proposed method of analysis for the particle distribution of sub-sieve particulate materials by the Society's Committee E-1 on Methods of Testing[2] is a good step in this direction.

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I (1933); also published in pamphlet form.

[2] Proposed Method of Analysis for the Particle Size Distribution of Sub-Sieve Size Particulate Substances, with Comments, Report of Committee E-1, Appendix III, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 919 (1930).

2. From a standpoint of facilitating the construction of graphs to show the shape of the various frequency distributions used in particle size work, there is an advantage in using class intervals of measurement that are equal or that increase in a geometric ratio with particle size. The latter is the more practical in most particle size work. If, however, irregular class intervals are used in measurement, the shape of the distribution can be shown by plotting "per cent per unit size" as in Fig. 1.

3. No single function gives much information about the original distribution. Two or more specific functions such as average diameter, standard deviation and relative percentage give helpful information as to the shape of the distribution. If the above factors are given proper consideration, the presentation of fineness data bears some real weight.

Past considerations in particle size work have been given largely to a description of the numerical results obtained from a single sample and from one technique or method of analysis. In the problem of discovering the frequency distribution of particle size for a material, it is necessary to give consideration to the variations of observed results obtained from several samples and for different methods of analysis, if one is to determine how much of the observed variability is attributable to the material itself and how much is due to variations of measurement.

It is the authors' experience that no single functional form can be used to describe all types of distributions. Examples cited in this paper do not have similar distributions as shown by the graphs.

*Acknowledgment.*—The authors wish to express thanks to Messrs. L. T. Work of Columbia University, G. O. Hiers of the National Lead Co., H. F. Dodge and H. G. Romig of the Bell Telephone Laboratories for helpful suggestions and criticisms in the preparation of this paper.

# METHOD AND APPARATUS FOR THE RECOVERY OF ASPHALT

By Gene Abson[1]

## Synopsis

This paper describes a method and the form of apparatus for use in recovering bitumens, particularly asphalts, after they have been extracted from the structures in which they were originally used. In pavements this means the extraction and recovery from the mineral matter with which the asphalt has been mixed. The specific method is that the asphalts are extracted with benzol and the benzol then removed by distillation in an atmosphere of carbon dioxide. The apparatus is simple, comprising a 250-ml. wide-mouth extraction flask fitted with a delivery tube, connected with a water-cooled condenser, heated in an oil bath. Through the stopper, in addition to the delivery tube, are inserted a thermometer and a copper tube, perforated to admit carbon dioxide gas at controllable rates, resting on the bottom of the flask.

One of the important features of this method is that relatively low distillation temperatures are required for complete removal of the solvent. This, then, permits the recovery of the bitumens in the exact state in which they existed prior to extraction. Former methods either did not or could not use benzol, and if benzol were used, the temperatures required produced hardening during the distillation. Other former methods were so involved or required so long a time for completion that they are impracticable in commercial operations.

The experimental data in this paper cover a range of asphaltic materials such as are commonly used in paving and other industries, from extremely soft to extremely hard. The tabulated data show that the method has a wide range of applicability, even using bitumens of relatively low volatility.

## Introduction

The usual method for extracting asphalt from the products or structures in which it is incorporated is by means of a solvent. Oftentimes, following the extraction, it is desirable to separate the asphalt from the solvent in order to render the asphalt available for examination or study. Any recovery system for this purpose must be applicable over a wide range of asphaltic products, since in most cases no prior knowledge is available concerning the type or grade of materials originally used, except that experience will indicate its relative degree of hardness or softness. Furthermore, the recovery method must be so conducted that the solvent is completely removed without causing any hardening or polymerization by excessive temperature or prolonged time of heating and that no other significant or material alteration in physical properties takes place during the recovery operation. Previous methods described in the literature are more or less uncertain, are restricted to the use of carbon disulfide, or are too tedious and time-consuming for commercial adaptation.

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Dow[1] describes a method of evaporation in which carbon disulfide must be used as the solvent. Bateman and Delp[2] and Bateman and Lehmann[3] describe a distillation test under a partial vacuum of 180 mm. of mercury at a temperature of 500 F. (260 C.) for removing carbon disulfide used as the solvent. Wilson[4] outlines an involved method of distillation, water-bath treatment, boiling-water-bath treatment, and an oil-bath treatment, of over 16 hr. duration and, finally, he is restricted to the use of carbon disulfide. Walczak and Rice[5] describe a purely mechanical method for the recovery of fluid or semifluid tars but which we have found is not applicable to the removal of semisolid or solid asphaltic materials. Greutert[6] describes a method in which distillation is conducted in the first stage with the aid of carbon dioxide, and in the last stages with a vacuum of 7 to 12 mm. at a temperature of approximately 100 C. above the softening point of the asphalt under treatment. This, therefore, presupposes a rather definite knowledge of the characteristics of the asphalt under recovery. Greutert also refers to an article by Professor Suida[7], in which he describes a method of distillation under vacuum for vaporizing the major portion of the solvent and the removal of the last traces by agitation or stirring under a bell jar in an atmosphere of carbon dioxide. Greutert further points out that asphaltic solutions harden when allowed to stand for longer than 24 hr.

The A.S.T.M. Tentative Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc or Mineral Granules, also Asphalt Shingles Surfaced with Mineral Granules (D 228 - 31 T),[8] outlines a procedure for the recovery of bituminous material used in saturating felt. In this method the solvent may be benzol, in which case the distillation is carried out at a temperature of 275 F. (135 C.) under a vacuum of 0.5 in. of mercury maintained for one hour. Aside from the fact that a sufficient amount of material for a penetration test cannot be readily obtained using the 200-ml. flask specified, the method is extremely tedious and requires hours of constant and attentive observation with proper manipulation of the vacuum apparatus. Experimental tests with asphalts of known penetration have shown deviations of 10 per cent or more in penetration. Without further experimenting, this method cannot be considered as accurate in its present form. The use of vacuum distillation at higher temperatures, particularly as specified in some of the preceding references, has, in our experience, always resulted in considerable hardening of the asphalt.

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[1] A. W. Dow, "The Testing of Bitumens for Paving Purposes," *Proceedings*, Am. Soc. Testing Mats., Vol. III, p. 349 (1903).

[2] J. H. Bateman and Charles Delp, "The Recovery and Examination of the Asphalt in Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 465 (1927).

[3] J. H. Bateman and H. L. Lehmann, "The Effect of the Mixing Temperature on the Physical Properties of an Oil Asphalt in a Sheet Asphalt Mixture," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 943 (1929).

[4] D. M. Wilson, "The Manufacture and Testing of Asphalt Paving Materials," *Roads and Road Construction*, Vol. 10, No. 111, p. 72 (1932).

[5] M. R. Walczak and H. I. Rice, "The Extraction of Bitumens from Mineral Aggregates," *Industrial and Engineering Chemistry*, Vol. 12, No. 8, p. 738 (1920).

[6] J. Greutert, "The Recovery of Asphaltic Bitumens from Solutions as Far as Possible Without Altering Their Properties," *Journal*, Inst. Petroleum Technologists, Vol. 18, No. 108, p. 846 (1932).

[7] Suida, *Asphalt und Teer*, p. 197 (1931).

[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 818 (1931); also 1932 Book of A.S.T.M. Tentative Standards, p. 745.

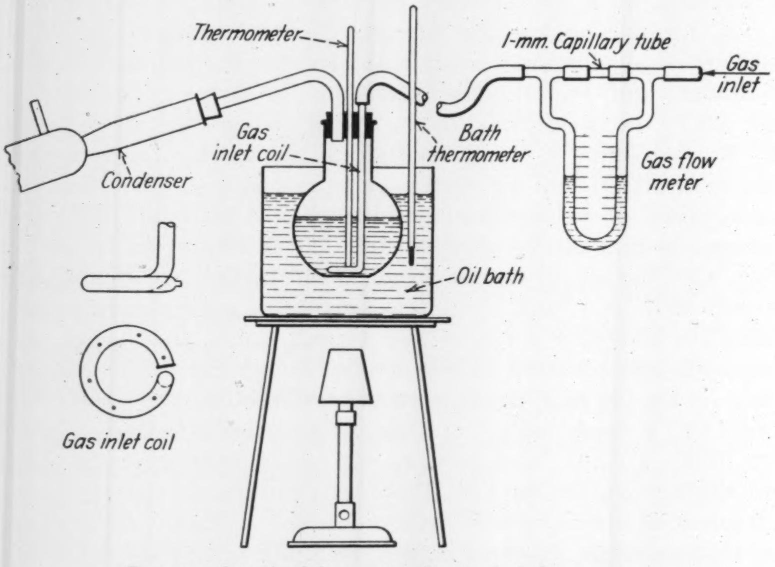

FIG. 1.—Set-up of Apparatus for Asphalt Recovery.

It has long been felt that benzol is a more desirable solvent for ordinary routine laboratory use, mainly because of the high vapor pressure and flammability of carbon disulfide, and that a method of removing the benzol for further examination of the bitumen would be welcomed generally. In fact, Bateman and Lehmann[1] say, "Careful consideration was given in this work to the use of other solvents and a large amount of work was done with benzol. We found that we could not obtain consistent results with benzol extractions . . . For some reason, which we are unable to explain, benzol consistently could not be removed as well as carbon disulfide. We, therefore, abandoned its use."

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[1] J. H. Bateman and H. L. Lehmann, "The Effect of the Mixing Temperature on the Physical Properties of an Oil Asphalt in a Sheet Asphalt Mixture," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, pp. 947–948 (1929).

A method has been devised which quickly and easily permits the recovery of asphalts from benzol solutions at a maximum distillation temperature of 300 to 325 F. (150 to 163 C.) and with an accuracy within the A.S.T.M. range for tolerance for penetration, softening point and similar tests. It is applicable, with this degree of accuracy, to asphalts as soft as 150 penetration at normal temperature; as high as 200 F. (95 C.) in softening point, and as low as 350 F. (175 C.) in flash point.

## TESTING APPARATUS

With the apparatus shown in Fig. 1, the operation comprises essentially an ordinary distillation in the presence of an inert gas, which in this case is carbon dioxide, and is similar in all respects to steam distillation without the troublesome condensation of vapors to water with its accompanying irritation both to the material and the operator. Briefly, the apparatus comprises a 250-ml. wide-mouth extraction flask, fitted with a gooseneck vapor line made of approximately 10-mm. inside diameter Pyrex tubing and connected with a condenser. The gas inlet is an ordinary copper tube of approximately 5 mm. inside diameter, bent so that the lower end forms a circle of slightly less diameter than the mouth of the flask and at right angles to the length of the tube. The other end is fitted through the cork and connected by rubber tubing to the source of the gas. The copper coil rests upon the bottom of the flask and is perforated with $\frac{1}{32}$-in. holes along the bottom so that the entering gas is directed downward, giving optimum distribution and contact with the asphaltic solution. A thermometer is inserted through the same cork and is placed with the lower end of the bulb about $\frac{1}{4}$ in. from the bottom of the flask and in the center of the circle of the copper tubing, completing the assembly. The flask is then immersed up to the neck in an oil bath, the use of the oil bath being necessary to prevent foaming during the distillation.

## GAS AND SOLVENT

The gas used may be any one that is inert toward asphalts, but since carbon dioxide is readily procurable in cylinders, 99.9 per cent pure, and is neither harmful nor dangerous, it is preferred. The amount of gas used is measured by means of a simple flow meter which consists of an ordinary differential gage or manometer connected by means of tees to a capillary tube. The particular set-up used in this work comprises a manometer of 5-mm. glass tubing connected by rubber tubing with a 1-mm. capillary tube, colored water being the gage fluid. The differential in pressure is due simply to the increased velocity through the restricted opening and is an application of a well-known principle of physics. The gas is drawn from the cylinder through a pressure regulating valve and is bled from this valve through a needle valve and connected by rubber tubing to the flow meter. The flow meter is easily calibrated by displacement of water, for which an ordinary 5-gal. water bottle may be used, connected with a manometer tube for pressure regulation and correcting for the vapor tension of the water. A graph may be constructed showing the rate of flow in cubic centimeters per minute for any gage reading measured in centimeters or inches. The various sizes of tubing used in the flow meter were selected primarily because they happened to be handy in the laboratory; any other convenient apparatus can be used.

The function of the gas is exactly the same as in any other distillation with immiscible vapors. Both by the reduction in partial pressure (Dalton's Law) and the mechanical agitation and sweeping effect of the gas, the solvent is more readily and completely removed at a lower temperature. In the former respect, the lowering of the boiling point is equivalent to distillation under reduced pressure, but the agitating effect materially reduces the time required for complete removal of the solvent; furthermore, after the first introduction of the gas, the trouble encountered with foaming is greatly reduced in comparison with vacuum distillation.

The benzol used for dissolving the asphalt need not be the chemically pure grade but should have a close-cut distillation range. It has been found that the Barrett Company's "Industrial Pure Benzol," having an initial boiling point of 174 F. (79 C.) and an end point of 178 F. (82 C.) is entirely satisfactory. Ordinary 90 per cent benzol, fractionated through a laboratory Hempel column to similate the above distillation range, may also be used.

## PROCEDURE

From 50 to 75 g. of the asphalt are either dissolved in benzol or extracted from the structure in which it was used, and distilled without the use of carbon dioxide until most of the benzol is removed. The copper tube is plugged with a cork to prevent syphoning prior to the introduction of the gas. When the inside temperature reaches

300 F. (150 C.), gas is admitted slowly, about 150 cu. cm. per min., corresponding to 0.5 cm. on the flow meter, for only a few seconds at a time. This is repeated at 3 to 5 F. (1.5 to 3 C.) intervals and thus serves to agitate the contents of the flask and minimize any foaming which might occur if the full quantity of gas were used at the start. When the inside temperature reaches 325 F. (163 C.), the gas is admitted at the rate of 875 cu. cm. per min., corresponding to 5cm. flow meter reading, and held for exactly 15 min. In order to maintain the temperature of the asphalt at 325 F. (163 C.), it is necessary to keep the temperature of the oil bath about 20 to 25 F. (11 to 14 C.) higher, due to the cooling effect of the gas. At the end of 15 min. the asphalt is ready for removal and may be poured directly into testing molds or tins. Sometimes with asphalts of the higher melting-point range, gas may be occluded, but this may be readily removed by heating to a somewhat higher temperature with slight stirring.

TABLE I.—ANALYSIS OF STEAM-REFINED MEXICAN ASPHALT.

| | ORIGINAL MATERIAL | COMPOSITE OF FIVE RECOVERED SAMPLES |
|---|---|---|
| Penetration at 77 F. (25 C.), 100 g., 5 sec | 61 | 61 |
| Penetration at 32 F. (0 C.), 200 g., 60 sec | 23 | 22 |
| Ductility at 77 F. (25 C.), 5 cm. per 60 sec | 145+ | 145+ |
| Cementing value at 41 F. (5 C.), kg-m | 0.15 | 0.16 |
| Elongation at 41 F. (5 C.), cm | 12+ | 12+ |
| Softening point, ring-and-ball method | 127 F. (53 C.) | 126 F. (52 C.) |
| Specific gravity at 77 F. (25 C.) | 1.044 | 1.043 |
| Flash point, Cleveland open cup | 545 F. (285 C.) | 540 F. (282 C.) |
| Loss, 5 hr. at 325 F. (163 C.), per cent | 0.02 | 0.06 |
| Penetration residue | 55 | 53 |
| Loss in penetration, per cent | 9.8 | 13.1 |
| Fixed carbon, per cent | 14.7 | 14.9 |

The selection of the above-stated rate of gas flow, time and temperature, while desirable, is entirely arbitrary and was ascertained purely by trial and error. The gas may vary plus or minus 100 cu. cm. per min., the time may vary plus or minus 2 min., and the control temperature may vary plus or minus 5 F. (3 C.) without any harmful effect upon the asphalt, and still insure complete removal of the solvent. A maximum temperature of 300 F. (150 C.) is more desirable for materials of 100 penetration or softer, but unless this can be previously ascertained, 325 F. (163 C.) may be used safely; likewise 350 F. (175 C.) would be more desirable for asphalts with a softening point of over 150 F. (65 C.).

As examples of the accuracy of this method as well as to illustrate the tolerances permissible in distillation time, and quantity of gas used, the following tables are shown. Table I shows a complete analysis of steam-refined Mexican asphalt both in its original state and after recovery from benzol solutions. Five individual batches of 75 g. each were recovered at a temperature of 325 F. (163 C.), using 5 cm. of carbon dioxide gas for 15 min. The penetrations of the five recovered samples were 61, 61, 61, 60 and 61.

TABLE II.—MEXICAN ASPHALT RECOVERED AT 325 F. (163 C.).

| ORIGINAL PENETRATION | RECOVERED PENETRATION | TEMPERATURE | TIME OF GAS, MIN. | AMOUNT OF GAS, CM. |
|---|---|---|---|---|
| 61 | 60 | 325 F. (163 C.) | 17 | 5 |
| 61 | 61 | 325 F. (163 C.) | 13 | 5 |
| 61 | 60 | 325 F. (163 C.) | 15 | 6 |
| 61 | 61 | 325 F. (163 C.) | 15 | 4 |

Table II shows the same asphalt recovered at 325 F. (163 C.) with time and amount of gas varied.

TABLE III.—ANALYSIS OF ASPHALTS.

| KIND OF ASPHALT | WEIGHT OF SAMPLE, G. | PENETRATION | | SOFTENING POINT, RING-AND-BALL METHOD | |
|---|---|---|---|---|---|
| | | ORIGINAL | RECOVERED | ORIGINAL | RECOVERED |
| California | 75 | 31 | 31 | | |
| | 50 | 31 | 31 | | |
| Mexican | 75 | 61 | 61 | | |
| | 50 | 61 | 61 | | |
| | 75 | 44 | 45 | | |
| | 50 | 44 | 43 | | |
| | 75 | 51 | 52 | | |
| | 50 | 51 | 51 | | |
| | 75 | 64 | 64 | | |
| | 50 | 64 | 62 | | |
| | 75 | 91 | 91 | | |
| | 50 | 91 | 91 | | |
| | 75 | 175 | 174 | | |
| | 50 | 175 | 171 | | |
| Smackover | 75 | 164 | 162 | | |
| | 50 | 164 | 159 | | |
| Mexican, air blown | 75 | 26 | 26 | 164 F. (73 C.) | 163 F. (73 C.) |
| Gulf coast, air blown | 75 | 27 | 27 | 197 F. (92 C.) | 198 F. (92 C.) |
| Bermudez | 75 | 47 | 46 | | |
| | 75 | 65 | 64 | | |
| Texaco | 75 | 52 | 53 | | |
| | 75 | 70 | 68 | | |

Table III shows a number of different brands of asphalts, both steam refined and air blown, of differing penetrations and softening points, recovered from benzol solutions at 325 F. (163 C.), using 5 cm. carbon dioxide gas for 15 min., using both 50- and 75-g. samples.

Another extremely vital requirement is that the asphalt solutions must be distilled on the same day on which they are dissolved. Considerable hardening takes place if the solutions are allowed to stand for more than 10 hr., and this is true with either benzol or carbon disulfide solvents. In some of the earlier data reported by other investigators, in which solutions were allowed to stand for as long as 72 hr., the accuracy of the results reported may have been due to the presence of small amounts of solvent not completely removed, which compensated for the hardening which undoubtedly must have taken place. Complete exploration of the rates of hardening in solution and the causes thereof has not been possible on account of the time required, even though this investigation was started over a year ago. It is being pursued and the results will be available for publication later. It can be definitely said, however, that almost all of the varieties of asphalt made from various crude oils by various processes show this same effect, although the rate and degree of hardening are not constant or uniform.

TABLE IV.—RESULTS OF HARDENING IN SOLUTION.

| WEIGHT OF SAMPLE, G. | TIME STANDING IN BENZOL, HR. | PENETRATION ORIGINAL | PENETRATION RECOVERED |
|---|---|---|---|
| 75 | 24 | 156 | 147 |
| 75 | 72 | 156 | 131 |
| 75 | 144 | 156 | 115 |
| 75 | 240 | 156 | 110 |
| 75 | 384 | 156 | 108 |
| 75 | 480 | 156 | 109 |

Table IV shows the results of hardening in solution of one kind of asphalt dissolved in benzol and recovered at a temperature of 300 F. (150 C.), using 5 cm. of carbon dioxide gas for 15 min.

## CONCLUSION

It has been shown that asphalts of widely varying degrees of consistency may be recovered from benzol solutions in substantially the identical physical state existing before such solution. It has been shown that benzol may be used as the solvent and that its complete removal is possible with the apparatus and method described. Of paramount importance is the fact that asphalts undergo some physico-chemical change if allowed to stand in a dissolved state for as long as 10 hr. and that accurate testing, therefore, requires removal of the solvent within this period. This has been found to be true whether the asphalts are dissolved hot or at room temperature.

# DISCUSSION

Mr. M. H. Ulman.[1]—The Committee on Materials of the American Association of State Highway Officials has done considerable work on the recovery of bitumen extracted from bituminous materials. In this research work a method has been developed in which it has been found that the asphalt cement can be recovered so that it checks within one point penetration of the original asphalt cement. This method utilizes a less involved apparatus than that described by Mr. Abson, and it has also been found in this investigation that benzol is the preferable solvent for such recovery testing. This committee, however, did not investigate the effect on consistency of the time element in consideration of the solvent action with benzol on the asphalt cement. Benzol was added to the asphalt cement at approximately four o'clock in the afternoon and the recovery started the following morning.

The straight distillation method was used utilizing a 2-l. round-bottom flask in which was incorporated 500 g. of asphalt cement and 900 ml. of benzol. The temperature at distillation was not permitted to exceed 500 F. (260 C.). The residue after distillation was cooled to 400 F. (205 C.) and the flask rotated and the contents poured into a large casserole. There was then added approximately 2 ml. of distilled water and a quick steam distillation effected by heating the contents in the casserole over a flame, and the material stirred while being heated. This procedure was repeated twice.

In order to obtain accurate and concordant results it is necessary to effect this quick steam distillation within a maximum time of ten minutes. A considerable hardening effect is obtained on the asphalt cement if the time in the quick steam distillation exceeds ten minutes.

I should be interested to know whether Mr. Abson has used his method in recovery of asphalt cement from paving mixtures in order to determine the effect on the consistency of asphalt cement by the heating of the bitumen and the mineral aggregates in the preparation of the road mixtures.

Mr. Gene Abson.[2]—No work has been done on determining what happens in a paving mixture. There is no doubt that asphalts do harden during the time they undergo mixing. That would show up under any kind of test that was at all satisfactory.

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[1] Assistant Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

[2] Consulting Chemical Engineer, Chicago Testing Laboratory, Inc., Chicago, Ill.

MR. A. W. DOW.[1]—While Mr. Abson has said that benzol is the best solvent, he does not mention the fact that carbon disulfide has a deleterious effect on some asphalts when they are extracted by it. Whether it is that it decomposes and the sulfur acts on the asphalt or whether it alters the solubility of the hydrocarbons in each other is unknown. Yet some asphalts are always less ductile after extraction with the carbon disulfide than with the benzol.

As to the method Mr. Ulman describes, we have used the benzol and the water in the residue, making an accelerated steam distillation, for many years. Possibly we have not done it as carefully as we should have, but we have not been very successful. This may be due to the fact that we have been trying to get results on lighter materials than the ordinary paving asphalt. I think that you will find in the report of the operation of the Engineer Highway Department of the District of Columbia in 1901 a description of that method written by myself.

I hope that Mr. Abson's method and also the method suggested by Mr. Ulman may be so developed that asphalt may be extracted from paving mixtures and similar materials without materially altering its characteristics. There is a great need for such a test.

MR. ULMAN.—In reference to Mr. Dow's remarks, our investigation was mainly conducted on asphalt cements of the consistency as generally used in paving work, that is between 50 and 100 penetration. We have not investigated the effect on the consistency in the recovery of softer asphalts.

The discordant results reported by Mr. Dow may have been occasioned by exceeding the maximum ten minutes distillation time.

I have a considerable amount of data on file based on cooperative tests made by various laboratories in which this method was used, and some very erratic results were obtained, particularly in those cases where the use of the so-called quick steam distillation was conducted over an hour.

MR. J. S. MILLER, JR.[2]—The difference in behavior between benzol and carbon disulfide in some work that Mr. Ebbert did several years ago in attempting to prove whether or not we had true solutions is pertinent; he found that carbon disulfide is probably not a true solvent for asphalt, while benzol is.

In our laboratory we have done a great deal of work distilling disulfide over linseed oil, attempting to remove all the free sulfur, and we finally decided on benzol as the solvent, which confirms the work that Mr. Dow and Mr. Abson refer to.

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[1] Chemical and Paving Engineer, New York City.

[2] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

MR. ABSON (*author's closure*).—I think that that loss in penetration after the 5-hr. test is about as close as you can obtain in trying to duplicate the result on the same material. In other words, there is a difference of two points in penetration, which is within the A.S.T.M. range of tolerance.

Penetration tests at 115 F. (46 C.) were not made because the material was just about at the limit of accurate penetration testing. The low temperature ductilities were not made, but the cementing value was substituted for that, and since that is a low-temperature test I think that it gives us the same information as a low-temperature ductility test would.

The two important tests, so far as indicating whether the material has undergone alteration or not, can be judged by the specific gravity and the fixed carbon, and they are just about as close as you can ever duplicate them. Those two tests will tell in a measure whether any alteration has taken place in the chemical properties of the asphalt. The susceptibility to temperature changes will apparently not be very greatly affected. The softening points are within 1 degree of each other, which is just as close as can be checked. The flash points are so close together it would prove almost conclusively that no benzol was present in the solution.

# A QUALITATIVE TEST FOR DETERMINING THE DEGREE OF HETEROGENEITY OF ASPHALTS

By G. L. Oliensis[1]

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## Synopsis

A test has been developed for determining qualitatively the degree of physical heterogeneity in asphalt. The test depends upon the precipitation that takes place when such asphalts are dissolved in or dispersed by 5.1 times their volume of certain petroleum naphthas. The term "carbenoids" is proposed as the name of the precipitated bodies.

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The heterogeneous character of bituminous substances has long been recognized. This heterogeneity is manifest even in hydrocarbon systems composed of members of the same series, as illustrated in the cold settling of Pennsylvania cylinder stock, which involves the precipitation of the heavy amorphous petrolatums from the paraffin hydrocarbons of lower molecular weight. It is also manifest in asphalts—in some to a greater degree than in others. For example, while the hydrocarbons in properly refined residuals from asphaltic crude oils show no tendency to separate, even after prolonged standing, the blown or oxidized residuals from the same crude oils are characterized by the gradual "sweating out" of an oily film on the surface and the slow development of a "cheesy" structure throughout the mass. Oxidized residuals from mixed-base crude oils show an even stronger tendency in the same direction; while blends of these with pressure-still residuals may be so unstable that when kept melted for only a few hours, a mushy, granular sediment may be precipitated on the bottom of the container.

According to Nellensteyn,[2] asphalt is a dispersion of micelles consisting of elementary carbon and adsorbed protective bodies in a medium of a mixture of hydrocarbons. It is conceivable that the nuclei of the micelles may consist not of elementary carbon, but of hydrocarbons having a sufficiently high carbon-to-hydrogen ratio to render them relatively insoluble in their adsorbed protective films. The micelles are part of an oleosol.

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[1] Laboratory, The Barber Asphalt Co., Madison, Ill.

[2] F. J. Nellensteyn, "The Constitution of Asphalt," *Journal*, Inst. Petroleum Technologists, Vol. 10, p. 211 (1924).

Laszlo Auer[1] has also described the structure of asphalt, rubber and similar hydrocarbons as an "iso-colloidal" system consisting of the highest molecular-weight components, surrounded by successive layers of hydrocarbons of decreasing molecular weight, all dispersed in a continuous phase consisting of the lighter hydrocarbons. He evidently assumes the entire system to be in a state of partial mutual solution, an equilibrium existing between solution and dispersion which permits an imperceptible gradation from the hydrocarbons having the highest carbon-to-hydrogen ratio to those having the lowest.

The addition of partial solvents which raise the interfacial tension between the micelles of Nellensteyn and the dispersing medium will tend to flocculate the former. Similarly the introduction of solvents to the system described by Auer will decrease the solvent power of the external phase and correspondingly increase the tendency to dispersion, with the result that the system may be rendered so heterogeneous as to precipitate the dispersed phase.

It appears that the lower the surface tension of the solvent, or the weaker its solvent power, or the greater the quantity of it added, the greater is the amount of flocculation caused and the wider the range of hydrocarbons affected. Carbon tetrachloride, having a relatively high solvent power, will precipitate a comparatively small amount of the highest molecular-weight hydrocarbons—the so-called "carbenes."[2] On the other hand, a large excess of a poor solvent such as petroleum naphtha will precipitate the maximum amount of hydrocarbons of a far wider range of molecular weight, including the "carbenes." This is illustrated by the naphtha-solubility test proposed by Richardson in 1892,[3] in which 100 ml. of petroleum naphtha is combined with but 1 g. of asphalt. The hydrocarbons thus precipitated are designated "asphaltenes" while the lighter hydrocarbons retained in solution are called "petrolenes." It has been found that each asphalt has its own characteristic "asphaltene" content.

As the amount of naphtha combined with an asphalt is reduced, the higher concentration of the petrolenes in the naphtha gives the latter a progressively greater solvent or dispersing power on the micelles, till a point is reached in the reduction of the naphtha where no flocculation at all results, as is illustrated in ordinary asphalt paints and cut-backs where the volume of the naphtha is seldom as much as twice that of the asphalt.

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[1] Laszlo Auer, "Colloid-Chemical Changes in Rubber and Fatty Oils," *Transactions*, Inst. Rubber Industry, Vol. 4, p. 499 (1929).

[2] Clifford Richardson and C. N. Forrest, "Carbon Tetrachloride and Its Use as a Solvent for Differentiating Bitumens," *Journal*, Soc. Chemical Industry, Vol. 24, p. 310 (1905).

[3] Clifford Richardson, "Petrolene or Bitumen Soluble in Petroleum Naphtha," Sixth Annual Report of the Inspector of Asphalts and Cements, Washington, D. C. (1891–92).

If, however, the amount of the naphtha is kept at some intermediate point, relatively near to that at which flocculation may be expected to begin, the significant discovery has been made that a wide divergence appears in different asphalts in respect to their tendency to flocculate. In some asphalts treated with this amount of naphtha the micelles remain permanently dispersed; in other asphalts they may remain dispersed for only a time—say a few hours or a few days—after which flocculation occurs; while in still other asphalts flocculation occurs immediately. These various asphalts may have all yielded exactly the same "asphaltene" content by the usual naphtha-solubility test.

It seems clear from the foregoing that in the first of the three types of asphalt discussed in the previous paragraph the micelles or asphaltenes must have been more truly compatible with their associated petrolenes than in either of the other types; in the second type the asphaltenes must have been less compatible, and in the third still less so. In other words, the first type of asphalt must have been relatively homogeneous, the second relatively heterogeneous, and the third, still more heterogeneous. Furthermore, the sooner flocculation develops, and the greater the amount of flocculation, the greater is the degree of heterogeneity indicated.

In this study of the heterogeneity of asphalts, the ratio of naphtha to asphalt can of course be varied over quite a range with consequent changes in results, but apparently a weight ratio of about 4 parts of naphtha to 1 part of an asphalt having a specific gravity of one gives reproducible qualitative indications of the degree of heterogeneity. In order to obviate the variable due to the specific gravity of the asphalt, a volume ratio of 5.1 parts of naphtha to 1 of asphalt has been adopted in this work. In a relatively homogeneous asphalt, the residue left by this ratio of solvent will be very small or nil, while, as the heterogeneity of the asphalt increases, the amount of residue will increase. For this residue we propose the name "carbenoids." The test described below gives a means of distinguishing between asphaltic products which might appear similar or identical in any of the usual naphtha solubility tests.

Naphthas from various sources have been tried in this test; those having the same general boiling range seem to give about the same result. Apparently a virgin type of naphtha (not a product of cracking) would be the most easily duplicated. However, such a naphtha from Smackover or similar crude oils seems to have greater solvent power than the others used and is not applicable to this test.

The naphtha so far found most desirable meets the following specification:

Straight run overhead free from cracked products of any kind.

| | |
|---|---|
| Gravity | 49 to 50 deg. A.P.I. |
| Initial boiling point | about 300 F. (150 C.) |
| End point | about 400 F. (205 C.) |

The aniline number may be a means of further controlling the character of the naphtha to be employed.

The rapidity with which the asphalt is dispersed in the naphtha is an important factor in obtaining concordant results; and since rapidity of dispersion depends upon first, the temperature maintained during dispersion, second, the degree of agitation and third, the time required, it has been found necessary to control these factors in making this test. In conducting the test in our laboratory, a weight of asphalt equivalent to 2 ml. is placed in a weighed Erlenmeyer flask of 25-ml. capacity. If the asphalt does not flow readily at atmospheric temperatures it is cautiously heated in the flask on a hot plate until it flows and spreads over the bottom. After cooling, 10.2 ml. of cold petroleum naphtha are added and the flask and contents weighed. A stopper, in which is fitted an 8-in. length of open $\frac{1}{4}$-in. glass tubing, is inserted in the neck of the flask. The latter is immediately immersed in a glycerine bath maintained uniformly at a temperature 200 F. (110 C.) higher than the melting point of the asphalt, though if the asphalt is a thin liquid or cut-back heating is unnecessary. The degree of agitation is limited to a vigorous swirling of the contents of the flask with a rapid circular motion for 5 sec., immediately after adding the naphtha and similarly at the end of each minute thereafter until complete dispersion has been obtained. Dispersion should be completed in 6 to 8 min.; if not, the test should be repeated with the bath maintained at a 25 F. (14 C.) higher or lower temperature until dispersion is effected within the stipulated period of time. As soon as complete dispersion takes place the flask is cooled to room temperature, without chilling below that temperature, and any loss is made up by the addition of fresh naphtha.

A drop of the mixture is placed upon a clean dustless glass plate inclined at an angle of about 45 deg. If the drop on flowing out gives an even, smooth, clear, glossy, brown film after evaporation of the solvent, the mixture should be set aside at room temperature for 24 hr., in its tightly stoppered flask. At the end of this time the mixture is vigorously stirred until uniform and then retested. If the film obtained is again smooth and clear after evaporation of the solvent, the asphalt is classified as homogeneous. However, should it be observed either in the case of the freshly prepared mixture or after it has been standing 24 hr. that as the drop flows there develops in the center of its path a dull, matte streak, well defined against the smooth, glossy, light-brown outer zone, the asphalt is classified as heterogeneous.

Instead of placing a drop of the mixture on an inclined glass plate it may be allowed to fall on a filter paper. When the latter is used, relatively homogeneous asphalts give a uniformly brown circular stain, while relatively heterogeneous bitumens give a brown stain with a much darker spot, which appears rough, in the center. Where but small amounts of "carbenoids" are present, they may fail to develop this characteristic rough nucleus on filter paper as clearly as on glass; therefore, the "spot" test on filter paper is not quite as delicate as on glass, though less subject to variations in personal interpretation.

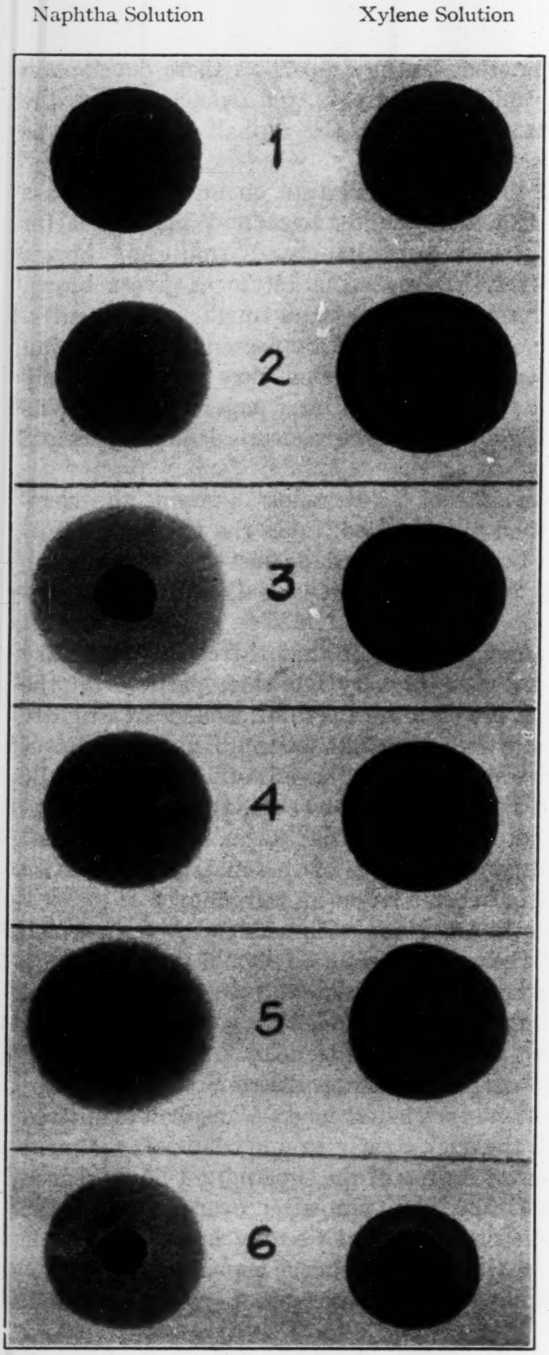
With very minute amounts of "carbenoids" present, the characteristic nucleus, even when glass is used, takes the form of a faint, fine hazy area that can best be detected against the surrounding clear zone by observing the glass against a dark background by reflected light.

Sometimes the haze may be so faint that there is a possibility it is due merely to the presence of very fine dust particles in the solution. If doubt exists, it is well to set the solution aside in a tightly sealed container and test, after thorough stirring, at intervals of about a week. If after several weeks the haze is still slight it can be considered as due to dust particles. If, however, the haze becomes gradually more pronounced, it is evident that traces of "carbenoids" are present in the asphalt, since particles of this material, like those of most precipitates, tend to become larger on standing.

The nucleus may also be examined with a microscope, in which case a very small drop of the naphtha solution should be placed on a microscope slide held in a horizontal position and the solvent allowed to evaporate. A dark rough spot surrounded by a clear glossy ring is formed when the asphalt displays heterogeneity. When magnified the rough spot is found to contain numerous discrete particles.

When a naphtha solution of a "heterogeneous" asphalt is allowed to stand undisturbed with the flask tightly stoppered, a soft sediment slowly settles to the bottom. A drop of the supernatant liquid placed on a glass plate gives a clear glossy film after evaporation of the solvent. The color of this film will be noticeably lighter than that obtained when testing a relatively homogeneous asphalt.

With asphalts containing fine non-bituminous matter, a dull,



No. 1. Carefully steam-refined residual.

No. 2. Pipe still residual (subjected to local overheating).

No. 3. Cracking coil residual.

No. 4. Blend of 100 parts of No. 1 and 50 parts of No. 2.

No. 5. Blend of 100 parts of No. 1 and 33 parts of No. 2.

No. 6. Asphalt containing fine non-bituminous matter.

FIG. 1.—Results Obtained with the Heterogeneity Test on Several Kinds of Asphalt.

central streak is always obtained. Since the "carbenoids" of a "heterogeneous" asphalt are soluble in or dispersed by xylene, this solvent offers a means of distinguishing between "carbenoids" and such fine non-bituminous matter. If the gritty, dull streak is obtained with xylene as well as with naphtha, the original streak was caused by the presence of the non-bituminous matter; but if the streak does not appear using xylene, "carbenoids" are present in the asphalt.

Figure 1 illustrates the results obtained with the "heterogeneity test" on several different kinds of asphalt, using both naphtha and xylene as the solvent.

Experiments over a 6-year period have established that by this test the following types of bitumens are classified as homogeneous:

1. Steam-refined residuals known to have been refined without serious cracking.
2. The bitumen of certain native asphalts.
3. Some types of slightly oxidized residuals from asphaltic-base crude oils.

Types of bitumens which by this test are classified as heterogeneous are:

4. Steam-refined residuals that have been overheated during the refining operation.
5. Cracking-coil residuals.
6. Highly blown residuals.

Although all asphalts are more or less heterogeneous as shown by the recognized tests with partial solvents, the proposed test seems to afford a more delicate means for the comparison of the degree of heterogeneity of various asphalts. For many purposes a low degree of heterogeneity is desirable while in certain specific fields a higher degree of heterogeneity is indicated.

In the course of the employment of this test it has been applied to hundreds of samples derived from many different crudes, from a number of different refineries and manufactured by widely different processes, and the conclusions set forth in the paper appear amply supported by the data so far accumulated. Nevertheless additional exploratory investigations are at present under way, and it is the author's thought that by making this test immediately available, other laboratories may cooperate in this study in order to establish the full extent of its usefulness.

# DISCUSSION

MR. V. NICHOLSON[1] (*presented in written form*).—While it was early recognized in the study of asphalts, that they were composed of two main constituents, the first a comparatively solid material called asphaltene, and the second an oily material called petrolene, still the manner in which these two components are mixed had not been studied until recently. In order successfully to study the matter it became necessary to find out in what physical system asphalt belonged. It has been learned that asphalt does not belong to the same system as a solution of salt in water, of sugar in water, of gelatin in water, or a suspension of carbon in petroleum oil, but is like a protected suspension of solid or liquid in water brought about by the use of a protective colloid.

Thus Nellensteyn[2] has pointed out that asphalt is a protected lyophobe sol, in which system certain hydrocarbons (the asphaltenes) are protected or surrounded by the so-called asphaltic resins, and then dispersed in an oily medium of the nature of heavy lubricating oil. The oil-loving nature of the protecting asphaltic resins makes the colloid take on the character of a lyophilic system.

Nellensteyn has pointed out that the interfacial tension between the dispersed particles and its dispersing medium is the dominating factor that determines the stability of the colloidal system.

It is easy to see that if asphalt is a two-phase system and we add a solvent which is miscible with the oily medium but does not dissolve the disperse phase, we still have only a two-phase system.

It will be recalled from my paper on "Adhesion Tension"[3] that adhesion tension $A.T = S_1 - S_{12}$; where $S_1$ and $S_{12}$ are, respectively, surface tension of the solid or dispersed phase, and interfacial tension between this phase and that of the dispersion medium. According to Antonow's rule the interfacial tension $S_{12} = S_1 - S_2$; where $S_2$ is the surface tension of the dispersion medium. Substituting this value in the above equation the adhesion tension $A.T. = S_1 - (S_1 - S_2)$ or is equal to $S_2$. If this rule were always correct the adhesion tension would always be equal to the surface tension of the medium surrounding the dispersed phase which, of course, is not in conformity with the data established by experiment. While the strict interpretation of Antonow's rule is not correct except in ideal cases, still with a series of solvents from a chemically homologous series of hydrocarbon petroleums, the solvents will precipitate out the dispersed phase in the order of their surface tensions, because the interfacial tension between the solid and the liquid increases as the molecular weight of the liquid decreases.

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[1] Engineering Chemist, Bureau of Streets, Chicago, Ill.

[2] F. J. Nellensteyn, "Asphalt," Colloid Symposium, Vol. 3, p. 537.

[3] V. Nicholson, "Adhesion Tension," *Roads and Streets*," April, 1932.

Therefore, if we fractionate a chemically homologous petroleum such as that obtained in Pennsylvania or in certain places in Oklahoma, we can obtain a series of solvents with differing boiling points, specific gravities and surface tensions from practically the same chemical series.

On adding these solvents in uniform amounts to a given amount of sample of the various asphalts, we can determine what solvent will cause the characteristic test for heterogeneity. It will be recognized, of course, that in doing this we are altering the surface tension of the medium and its interfacial tension against the solid in proportion to the amount and surface tension of the solvent added.

TABLE I.—RESULTS OF SPOT TESTS FOR OVERHEATED RESIDUES, USING VARIOUS SOLVENTS.[a]

2 g. of asphalt, 8 g. of solvent.

| | Asphalt No. 1 | Asphalt No. 2 | Asphalt No. 5 | Asphalt No. 6 | Asphalt No. 7 | Asphalt No. 8 | Asphalt No. 10 | Asphalt No. 13 | Asphalt No. 14 | Asphalt No. 15 | Asphalt No. 18A | Asphalt No. 18B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Kerosine | Yes | No | No | No | No | No | No | No | No | No | Yes | No |
| Wadsworth-Howland solvent | Yes | No | No | No | No | No | No | No | No | No | Yes | No |
| Barber solvent | Yes | No | No | No | No | No | No | No | No | No | Yes | No |
| Gasoline | Yes | No | No | No | No | No | No | Yes | No | No | Yes | No |
| Skellysolve-S | Yes | No | No | No | No | No | No | Yes | No | No | Yes | No |
| Skellysolve-E | Yes | Yes | No | No | No | Yes | No | Yes | No | No | Yes | No |
| Skellysolve-C | Yes | Yes | No | No | No | Yes | Yes | Yes | Yes | Yes | Yes | No |
| Skellysolve-B | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes |

[a] "Yes" indicates presence of spot. "No" indicates absence of spot.

The accompanying Table I shows the results obtained using the filter paper spot test, with 2 g. of asphalt and 8 g. of solvent. The kerosine was the highest in surface tension, with the Wadsworth-Howland solvent, Barber solvent and gasoline very close together, followed by the series of solvents from the Skelly Oil Co., in the order of their decreasing boiling point ranges and surface tensions.

As will be seen in the table, samples Nos. 1 and 18A gave the heterogeneity test in all the solvents used, while No. 13 showed this characteristic in solvents having surface tensions up to and including that of gasoline. Nos. 5, 6, 7 and 18B showed this characteristic only on using the very light solvent. Samples Nos. 14 and 15 showed this reaction in next to lowest and the lowest boiling solvent.

All of the above tests were run by weighing out 2 g. of asphalt in a beaker flask, heating on a hot plate to soften the bitumen, and then accurately weighing in the 8 g. of solvent selected. Beaker flask with sample and solvent were then rapidly heated for a period not exceeding 2 minutes on the electric hot plate, and the asphalt brought into solution or dispersion by rapidly rotating the flask. When asphalt was in solution or dispersion, it was removed from the plate, replaced on the scale pan and any loss of solvent by evaporation on the hot plate made up by a further addition of solvent. The finished mixture was then allowed to cool to room temperature and the spot test made on a filter paper by dropping the liquid on it from a stirring rod. Where no spot test was shown, the solution diffused uniformly into the paper, which on drying out was uniform in color by transmitted light. These samples were then checked by placing a small amount of the liquid on a microscopic slide and then allowing the solvent to evaporate. The dry material on the slide was then examined by means of a microscope. Those samples that showed clear on the paper were clear on examination with the microscope; whereas those that showed a spot, showed characteristic spots in the surface, which were plainly discernible.

TABLE II.—SPOT TESTS ON ASPHALT SAMPLE NO. 13, USING VARYING QUANTITIES OF SOLVENT.[a]

| Amount of Solvent per 2 g. of Asphalt Sample | Clean Sample | | | | | | Contaminated Sample | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Spot Test on Solution Immediately After Cooling | | Spot Test on Solution After Standing 1 Day | | Spot Test on Solution After Standing 4 Days | | Spot Test on Solution Immediately After Cooling | | Spot Test on Solution After Standing 1 Day | |
| | Barber Solvent | Kerosine Solvent | Barber Solvent | Kerosine Solvent | Barber Solvent | Kerosine Solvent | Barber Solvent | Kerosine Solvent | Barber Solvent | Kerosine Solvent |
| 6 g. | Clear | Clear | Clear | Clear | Clear | Clear | Clear | Clear | Clear | Ring |
| 8 g. | Clear | Clear | Ring | Clear | Ring | Ring | Ring | Clear | Ring | Ring |
| 10 g. | Ring | Clear | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring |
| 12 g. | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring |
| 14 g. | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring | Ring |

[a] "Clear" indicates absence of spot. "Ring" indicates presence of spot.

In order to show the effect of the amount of solvent used in proportion to the amount of asphalt, I made up a series of samples, using asphalt No. 13. The idea in this case was to find out if it was possible to make this asphalt show a spot test in the higher boiling, higher surface tension solvents by increasing the amounts of these added. It was also desired to find out the effect of surface impurities in the asphalt, such as would fall into an open sample can after many months' storage in the open room. Beside this, it was desired to find the effect of time of standing of the dissolved asphalt on the spot test.

The results of this series of tests are shown in the accompanying Table II.

It will be seen that on running the spot test on the freshly cooled solution, even though this sample did not show the characteristic ring using the ordinary amounts of Barber solvent, still by adding just two more grams of it, the spot is shown. In the case of kerosine, it can be seen that an additional 4 g. were required to bring about the same results. On allowing these samples to stand for a day and then testing, it is found that the sample showed the spot in the 8-g. addition of Barber solvent, where it had not done so on treating the fresh solution. The 10-g. addition of kerosine showed the spot where only the 12-g. addition had done it before. On allowing the solutions to stand for four days, the Barber solvent samples behave as they did after one day's storage, but in the case of kerosine shows a spot in the sample containing only 8 g. of this material. I believe the foregoing series of tests demonstrate the importance of the amount of solvent added and the length of time of standing of solution before the test is run.

In the case of asphalt No. 1, which showed marked evidence of heterogeneity, it is almost certain that the asphalt was made at a high temperature and high pressure. Sample No. 18A, which was supposed to have been a Mexican asphalt, either was made at too high temperatures or else had some overheated residue added to it. It might be pointed out at this time that No. 18B was the same brand of Mexican asphalt as No. 18A and that it still showed such a big difference in this test. This comparison shows the importance of testing all asphalt and not depending on origin or brand name for quality.

As to the cause for asphalts behaving differently in this test, we know that all colloids, of which asphalt is a typical example, are in vigorous Brownian movement as the temperature is increased. If in the method of manufacture the temperature is increased too much, and in addition high pressures are employed, the individual particles of the dispersed phase in the oily medium collide, agglomerate, and become coarser particles and then tend to settle out of the system during the refining. If they settle near a highly heated place they are overheated and thus the asphaltenes may change into a carbon, which shows a higher interfacial tension toward the dispersion medium than where this heat and pressure is not so intense. On adding the various solvents, it can be seen that the particles showing the higher interfacial tension toward the medium will have this medium more easily removed from them than from the majority of the other dispersed particles.

Regarding the utility of the heterogeneity test, I believe it is one of the most valuable tests that has been evolved for many years for the testing of asphalt.

In regard to the manipulation, it will, of course, be necessary to standardize on the exact procedure to be followed, as my results show. Thus the character and amounts of solvent to be added to the asphalt will have to be carefully standardized. As indicated by Mr. Oliensis, by adding a requirement for the solvent that it should show an aniline number of a certain temperature, in addition to its other characteristics already mentioned, I feel that the character of the solvent can be fixed.

I have inserted a clause in our specifications for both flux and asphalt, which states in effect that when a 15 per cent by volume solution of asphalt in Skellysolve-S is made, and a drop placed and allowed to dry on a microscope slide, the microscopic image shall not differ from that shown by the same concentration of asphalt in carbon tetrachloride. It can be seen from a comparison of proportions that I use a more dilute solution than Mr. Oliensis, but instead of passing judgment after one day, I pass judgment immediately after the sample has cooled and been placed on the microscope slide, dried, and examined.

MR. L. M. LAW[1] (*presented in written form*).—For many years practically all specifications for asphalt paving cements and road oils have included a requirement that "the material shall be homogeneous" in character, but so far as I am aware no laboratory test has ever been proposed for determining compliance with this feature. The appearance on the market in recent times of asphalts from new sources and processing methods has, however, made it highly desirable that this feature of homogeneity be given more serious consideration.

Accepting the view that the liquid asphaltic materials as found in nature are colloidal dispersions and the well-known fact that colloidal solutions are subject to alteration by heat, it seems reasonable to assume that excessive heat of processing, whether it be an unintentional local or general overheating or the deliberate application of extreme heat with pressure as in cracking, tends to render the dispersed phase coarser with consequent reduction of suspendability. The proposed test gives us an insight into this physical condition of asphaltic materials.

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[1] Asphalt Technologist, Shell Petroleum Corp., St. Louis, Mo.

Having been privileged to cooperate with Mr. Oliensis in the latter stage of his investigation, I am of the opinion that the "spot" test on paper should be of immediate interest in evaluating the liquid asphaltic road materials. In this connection, certain highway engineers have long expressed the need for a test that would differentiate straight refined products from those of pressure still origin so that both types could be better allocated to appropriate fields of usefulness. At present this is being attempted by laboratory evaporation to "asphalt contents" of specified penetration and then testing these residues for ductilities at arbitrary temperatures. The proposed homogeneity test appears to be a promising substitute for the cumbersome and less scientific procedure. As to the procedure based on observing the characteristics of a film on glass, there is, of course, no simple method for interpreting results so that this more delicate form of the test is scarcely adaptable for specification purposes.

Both forms of the test should be of particular concern to the asphalt producer since they afford a means of studying more intimately his refining process and determining more accurately the critical operating temperature for a specific raw material.

MR. C. M. BASKIN.[1]—It would seem to me that this type of test is very apt to be misinterpreted, aside from the fact that it would be very difficult to standardize. It is of interest to note that nearly all oxidized products may give adverse results by Mr. Oliensis' test and yet it does not mean that oxidized asphalts are not suitable materials.

We have had an opportunity to study the matter of uniformity or homogeneity of oxidized asphalts by filtration and stain tests and the results were not sufficiently convincing as either indicating quality or determining source.

I think that this matter should be given considerably more study. I am not certain that a test of this sort would even serve the purpose of identifying source of asphalt, if this is what Mr. Oliensis had in mind. We have made a number of tests along similar lines and Mr. Prévost Hubbard has also done some work along these lines, all of which would throw more light on it. Before making any definite conclusions in regard to value of Mr. Oliensis' determinations it would indeed be advisable to go into the subject of selective solubility more thoroughly.

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[1] Assistant Superintendent, Imperial Oil Refineries, Ltd., Montreal, P. Q., Canada.

MR. G. L. OLIENSIS[1] (*author's closure, by letter*).—I wish to clarify a few points concerning which there appears to have arisen some misunderstanding.

First, as to the difficulty Mr. Baskin points out of standardizing the test: The test was admittedly hard to standardize, both on account of the possible variations in the type of naphtha used and the many variables introduced by variations in the time, temperature, and degree of agitation employed in the blending operation. However, special pains were taken to make the test as exact and as rigidly controlled in every detail of procedure as years of experience with, and critical analysis of, the test could suggest. Limits were set for the type of naphtha, the size and shape of the container, the amount of asphalt and naphtha, the temperature of the solution, the rate of solution, the exact manner of agitation and the duration of the test.

Second, as to the possibility Mr. Baskin refers to of misinterpreting the significance of the test: I have tried from the start to avoid this by carefully weighing each statement made. This test has not been submitted as a test for quality. It will not serve to identify the geographical source of an asphalt—a fact Mr. Nicholson corroborates; nor will it necessarily identify the process used in refining. But it does reveal certain profound changes in structure caused among other things by excessive heat treatment or appreciable oxidation; and it therefore offers a new means for studying the internal structure of asphalts (except those that contain fine non-bituminous matter, whether mineral or carbonaceous or otherwise, the presence of which would mask the test and prevent drawing conclusions therefrom).

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[1] Laboratory, The Barber Asphalt Co., Madison, Ill.

# A STUDY OF ADSORPTION AS A METHOD FOR THE DETERMINATION OF THE SURFACE OF PULVERIZED COAL

By R. A. Sherman,[1] C. E. Irion,[2] and E. J. Rogers[3]

## Synopsis

This experimental investigation was undertaken to determine the possibility of the determination of the surface of pulverized coal by the amount of dye adsorbed. The literature shows that the pH value of the solution, the composition and structure of the dye, and the concentration of the dye are important factors. Preliminary experiments showed that methylene blue medicinal was a suitable dye that was adsorbed by coal, although the attainment of equilibrium was so slow as to indicate sorption.

Adsorption measurements were made on 11 sized samples ranging from No. 8 to 200 of each of three coals. When the total area of the coal is plotted against the total dye adsorbed a separate curve results for each coal. The results did not follow Freundlich's isotherm, but the results for two coals plotted as straight lines according to the equation $x = a - \frac{a}{s^k}$, where $s$ is surface, $x$ is the dye adsorbed, $a$ is the original amount of dye, and $k$ is a constant; the third coal did not follow this equation for the smaller sizes.

As the dye adsorbed per unit of surface was not the same for different coals, the practicability of the application of the method for the determination of surface in grindability studies is doubtful. The method has, however, interesting possibilities for the characteristics of different coal beds.

Pulverized coal, as prepared for combustion, normally has 40 to 90 per cent by weight of sizes that will pass the No. 200 sieve, the openings of which are 0.074 mm. square. The ordinary sieve analysis gives, therefore, no information as to the true size of the larger part of the coal. A similar condition exists in regard to cement, carbon black, zinc oxide, clays, and any fine powders whose properties depend on their surface.

A particular need for a rapid and accurate method for the determination of the mean size or total surface of the sub-sieve sizes of coal arose in connection with the investigation of the combustion of pulverized coal that is being conducted at the Battelle Memorial Institute.

A similar need is apparent in connection with research on the crushing characteristics, now commonly called the grindability of coal. Methods adopted for the determination of grindability are based on Rittinger's law, which states that the work done in crushing is proportional to the new surface exposed. The procedure reported by Hardgrove,[1] that is also used by others, consists essentially of the application of a given amount of work to a sample of given size and the determination of the new surface produced. The total surface is determined by screen sizing to the No. 200 or No. 325 sieve with the assumption of an arbitrary factor for the material that passes the smallest sieve used.

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[1] Fuel Engineer, Battelle Memorial Institute, Columbus, Ohio.

[2] Physical Chemist, Battelle Memorial Institute, Columbus, Ohio.

[3] Assistant Fuel Chemist, Battelle Memorial Institute, Columbus, Ohio.

Workers who have used this method report generally satisfactory agreement between the capacity developed by the mills or the power required to grind various coals and the laboratory results even with such assumptions, but it does not appear reasonable that coals of different hardness would have the same specific surface for the sub-sieve material.

Because it appeared important to prove or disprove whether such assumptions were justified, the Institute undertook a survey of the possible methods for the determination of the surface of finely ground materials and conducted an experimental investigation of the possibilities of the determination of surface of pulverized coal by adsorption.

## OTHER METHODS FOR SURFACE DETERMINATION

Some of the methods that have been used for the determination of the size of finely divided materials are: (1) Microscopic counting, (2) rate of solution, (3) sedimentation, (4) elutriation, and (5) adsorption. The merits of these methods have been reviewed by Roller,[2] who describes the construction, use, and results with an air analyzer or elutriator on cements and pigments. A later paper[3] presents further refinements and size analyses of a portland cement. Roller presents the air analyzer as an accurate and rapid apparatus for the measurement of particle size. The authors have tried, however, an apparatus described by Bouton and Pratt,[4] which is similar to but less refined than that used by Roller, and found that clean separation of particles of coal required an extremely long time, owing to the tendency of fine coal particles to adhere closely to larger ones.

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[1] R. M. Hardgrove, "Grindability of Coal," *Transactions*, Am. Soc. Mechanical Engrs., Fuels and Steam Power Division, Vol. 54, No. 1, p. 37 (1932).

[2] P. S. Roller, "Separation and Size Distribution of Microscopic Particles," U. S. Bureau of Mines *Technical Paper No. 490* (1931).

[3] P. S. Roller, "Measurement of Particle Size with an Accurate Air Analyzer: The Fineness and Particle-Size Distribution of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 607 (1932).

[4] C. M. Bouton and J. M. Pratt, "Rate of Combustion of Coal Dust Particles: Part I, Size Classification of Finely Powdered Coal in Air Currents," *Cooperative Bulletin No. 12*, U. S. Bureau of Mines and Carnegie Institute of Technology (1924).

## CHARACTERISTICS OF ADSORPTION

It is well known that powders will adsorb gases, liquids, and solutes from solutions. One of the principal workers on adsorption has been Paneth,[1] who has shown that the surface area of powders can be determined by measurement of the amount of dye or radioactive material adsorbed. Bancroft[2] showed that the amount of dye adsorbed is proportional to the concentration of the dye used and to the pH value of the dye solution. Rideal and others[3] have used the dye adsorption method to measure the surface of charcoal. Dye adsorption has been adopted as a tentative standard by the American Foundrymen's Association to test the quality of molding sand,[4] but no attempt is made to interpret the results in terms of area of surface.

The study of the literature showed that there was reasonable expectation that a relation could be found between the size or surface of pulverized coal and the amount of dye adsorbed by the coal from a dye solution. Having established this relation by the determination of the dye adsorbed on closely sized samples, it should be possible to place a given weight of any pulverized coal in a dye solution of given concentration and, when the amount of dye adsorbed was determined, to read from a chart the total surface or mean diameter of the sample. Whether or not the dye was adsorbed in a monomolecular layer would be of no importance so long as a definite relation between the surface and amount of dye adsorbed was established.

Several factors must be observed in the conduct of these experiments:

1. The amount of dye adsorbed is dependent on the pH value of the solution; therefore, this must be held constant, preferably on the alkaline side. A value of 8.0 was chosen.

2. The chemical composition of the solution is important. The concentration of all ions in solution must be controlled.

3. The composition and structure of the dye is of importance.

4. The amount of dye adsorbed is a function of the concentration. It would be desirable, therefore, to determine the dye adsorbed at an end point of constant concentration in all experiments.

## ACKNOWLEDGMENTS

Two Progress reports on this work have been submitted to Subcommittee VII on Pulverizing Characteristics of Coal, of the

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[1] F. Paneth and W. Vorwerk, *Zeitschrift für physicalische chemie*, Vol. 101, p. 445 (1922).

[2] W. D. Bancroft and C. E. Barnett, Colloid Symposium Monograph, Vol. 6, p. 73 (1928).

[3] E. K. Rideal, *Journal*, Chemical Soc., Vol. 129, p. 1813 (1925).

[4] American Foundrymen's Association, Testing and Grading of Foundry Sands, Standards and Tentative Standards, p. 104 (1931).

Society's Committee D-5 on Coal and Coke. Helpful suggestions have been made by members of that committee.

The search of the literature and the preliminary experiments were conducted by C. E. Irion, the experiments on Hocking coal to establish the method were carried on by E. J. Rogers, and the determination of the dye adsorbed by the Pocahontas and Illinois coals was made by C. E. Lucks. The preparation of the sized samples of coal and the microscopic examination and counting were done by L. F. Fairthorne.

## PRELIMINARY EXPERIMENTS

In a preliminary series of experiments, samples of pulverized coal, sized by screening, were placed in samples of dye solutions and sealed in glass tubes so that they could be shaken without contact of the dye with cork or other stoppers. The concentration of the dye remaining in solution was estimated by comparison with similar tubes containing known concentration of dye. These experiments showed:

1. That pulverized coal adsorbed all the five dyes tried: Naphthol Yellow *S*, Methyl Green, Ponceau *2R* (Red), Methylene Blue for Bacilli, and Methylene Blue Medicinal.
2. The addition of coal did not change appreciably the pH value of the solution.
3. A relation between the size of the sample and the amount of dye adsorbed was evident.
4. The dye adsorption was slow; about 10 hours was required to approach equilibrium. As surface adsorption is supposed to be practically an instantaneous process, the slow action pointed toward sorption, that is, the entrance of dye into cracks in the coal particles.
5. Because of the slow rate of the adsorption, no simple method could be conceived for the measurement of the adsorption at a fixed concentration of dye for all samples.

The results of these preliminary experiments gave enough promise that further work was done to determine definitely the value of the method.

## PREPARATION OF COAL SAMPLES

Adsorption experiments were conducted on three types of coal: (1) Hocking coal from the Ohio No. 6, or Middle Kittanning bed, (2) Pocahontas No. 3 coal, and (3) Illinois No. 6 coal from Jackson County, Ill. By repeated screening, 11 carefully sized samples of each of the three coals in the range of No. 8 to No. 200 were prepared. The samples were examined under the microscope to learn whether the separation was complete.

## APPARATUS AND METHOD

For the determination of the concentration of the dye remaining in solution a Bausch & Lomb Duboscq colorimeter was used. In this instrument light is passed through two solutions, one of known and the other of unknown strength; the depth of the solution of unknown concentration necessary to match the intensity of transmitted light through a given depth of the known solution is determined. The unknown concentration is calculated by Beer's law, which states that the intensity of the transmitted light is constant when the product of the thickness and concentration is constant.

TABLE I.—ADSORPTION OF METHYLENE BLUE BY HOCKING COAL.

Specific gravity = 1.42. Coal weight = 5 g. in 50 ml. of dye solution, 48 hours.
Dye concentration = 0.0010 g. per ml. pH value = 8.0.

| SIEVE | SIZE, MM. | MEAN DIAMETER, MM. | SPECIFIC SURFACE, SQ. CM. PER G. | TOTAL SURFACE, SQ. CM. | DYE, MG. | |
|---|---|---|---|---|---|---|
| | | | | | REMAINING | ADSORBED |
| No. 8 | 2.38 | | | | | |
| | | 1.88 | 22.47 | 112 | 45.05 | 4.95 |
| No. 16 | 1.19 | | | | | |
| | | 1.03 | 41.42 | 207 | 42.35 | 7.65 |
| No. 20 | 0.84 | | | | | |
| | | 0.726 | 58.20 | 291 | 41.00 | 9.00 |
| No. 30 | 0.59 | | | | | |
| | | 0.467 | 90.48 | 452 | 39.40 | 10.60 |
| No. 50 | 0.297 | | | | | |
| | | 0.273 | 155.00 | 775 | 38.10 | 11.90 |
| No. 60 | 0.246 | | | | | |
| | | 0.213 | 198.37 | 992 | 37.90 | 12.10 |
| No. 80 | 0.175 | | | | | |
| | | 0.162 | 260.82 | 1304 | 36.50 | 13.50 |
| No. 100 | 0.147 | | | | | |
| | | 0.136 | 310.69 | 1553 | 35.70 | 14.30 |
| No. 115 | 0.124 | | | | | |
| | | 0.114 | 370.64 | 1853 | 35.20 | 14.80 |
| No. 150 | 0.104 | | | | | |
| | | 0.097 | 435.60 | 2178 | 34.70 | 15.30 |
| No. 170 | 0.089 | | | | | |
| | | 0.082 | 515.29 | 2576 | 34.25 | 15.75 |
| No. 200 | 0.074 | | | | | |
| Mixed samples | | | | 368 | 41.0 | 9.0 |
| | | | | 1360 | 37.9 | 12.1 |
| | | | | 1893 | 36.8 | 13.2 |
| | | | | 7050[a] | 31.0 | 19.0 |
| | | | | 8125[a] | 31.3 | 18.7 |
| | | | | 8450[a] | 30.5 | 19.5 |

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[a] Area determined by microscopic counting.

Five-gram samples of coal, previously dried at 105 C., were placed in 50 ml. of dye solution of known concentration and fixed pH value adjusted to 8.0 to 8.2 by the addition of $NH_4OH$. At desired intervals a sample of about 10 ml. was removed for the determination of the concentration in the colorimeter.

TABLE II.—ADSORPTION OF METHYLENE BLUE BY POCAHONTAS COAL.

Specific gravity = 1.33. Coal weight = 5 g. in 50 ml. of dye solution, 48 hours.
Dye concentration = 0.0010 g. per ml. pH value = 8.0.

| Sieve | Size, mm. | Mean Diameter, mm. | Specific Surface, sq. cm. per g. | Total Surface, sq. cm. | Dye, mg. Remaining | Dye, mg. Adsorbed |
|---|---|---|---|---|---|---|
| No. 8 | 2.38 | | | | | |
| | | 1.88 | 24.00 | 120 | 46.80 | 3.20 |
| No. 16 | 1.19 | | | | | |
| | | 1.03 | 43.80 | 219 | 45.10 | 4.90 |
| No. 20 | 0.84 | | | | | |
| | | 0.726 | 62.14 | 311 | 45.10 | 4.90 |
| No. 30 | 0.59 | | | | | |
| | | 0.467 | 96.60 | 483 | 45.50 | 4.50 |
| No. 50 | 0.297 | | | | | |
| | | 0.273 | 165.25 | 826 | 44.70 | 5.30 |
| No. 60 | 0.246 | | | | | |
| | | 0.213 | 211.80 | 1059 | 43.90 | 6.10 |
| No. 80 | 0.175 | | | | | |
| | | 0.162 | 278.48 | 1392 | 43.90 | 6.10 |
| No. 100 | 0.147 | | | | | |
| | | 0.136 | 331.71 | 1659 | 44.30 | 5.70 |
| No. 115 | 0.124 | | | | | |
| | | 0.114 | 395.73 | 1979 | 43.50 | 6.50 |
| No. 150 | 0.104 | | | | | |
| | | 0.097 | 465.08 | 2325 | 43.10 | 6.90 |
| No. 170 | 0.089 | | | | | |
| | | 0.082 | 550.16 | 2751 | 42.40 | 7.60 |
| No. 200 | 0.074 | | | | | |

## RESULTS

In the first series of experiments duplicate samples of each of the 11 sizes of Hocking coal were placed in dye solutions of initial concentration of 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0 mg. per ml. The concentration of dye remaining in solution was determined at 12 and 24 hours and at 24-hour intervals up to 168 hours. Equilibrium was not established, even at 168 hours, but it was more nearly reached the higher the initial concentration of the dye.

The results also showed that the adsorption of dye did not increase regularly with increase of time as it should. This was probably due to a loss of dye in the removal and replacement of the sample removed from the colorimeter, and to change in the adsorption with change in the room temperature. Further experiments were accordingly run at constant temperature and readings were taken only once, at 48 hours. Although this was not the point of equilibrium, there should be a consistent relation among the adsorptions of the various sizes.

TABLE III.—ADSORPTION OF METHYLENE BLUE BY ILLINOIS COAL.

Specific gravity = 1.46. Coal weight = 5 g. in 50 ml. of dye solution, 48 hours.
Dye concentration = 0.0010 g. per ml. pH value = 8.0.

| SIEVE | SIZE, MM. | MEAN DIAMETER, MM. | SPECIFIC SURFACE, SQ. CM. PER G. | TOTAL SURFACE, SQ. CM. | DYE, MG. | |
|---|---|---|---|---|---|---|
| | | | | | REMAINING | ADSORBED |
| No. 8 | 2.38 | | | | | |
| | | 1.88 | 21.86 | 109 | 39.4 | 10.6 |
| No. 16 | 1.19 | | | | | |
| | | 1.03 | 39.90 | 200 | 37.9 | 12.1 |
| No. 20 | 0.84 | | | | | |
| | | 0.726 | 56.61 | 283 | 36.7 | 13.3 |
| No. 30 | 0.59 | | | | | |
| | | 0.467 | 88.00 | 440 | 35.5 | 14.5 |
| No. 50 | 0.297 | | | | | |
| | | 0.273 | 150.53 | 753 | 34.2 | 15.8 |
| No. 60 | 0.246 | | | | | |
| | | 0.213 | 192.93 | 965 | 32.5 | 17.5 |
| No. 80 | 0.175 | | | | | |
| | | 0.162 | 253.68 | 1268 | 31.4 | 18.6 |
| No. 100 | 0.147 | | | | | |
| | | 0.136 | 302.18 | 1511 | 30.2 | 19.8 |
| No. 115 | 0.124 | | | | | |
| | | 0.114 | 360.49 | 1803 | 27.5 | 22.5 |
| No. 150 | 0.104 | | | | | |
| | | 0.097 | 423.67 | 2118 | 26.7 | 23.3 |
| No. 170 | 0.089 | | | | | |
| | | 0.082 | 501.17 | 2506 | 24.0 | 26.0 |
| No. 200 | 0.074 | | | | | |

Tables I, II and III present the results of the determinations on the three coals. Three mixed samples and three samples of unscreened material were run with the Hocking coal. The mean diameter between the two sieve sizes was calculated as the square root of the mean of the squares of the limiting diameters. The area was calculated as if the particles were squares or cubes, area = $\frac{6}{\rho d}$ sq. cm. per g., where $\rho$ is the density and $d$ the mean diameter.

Figure 1 presents the results graphically where the total area of the sample is plotted against the total dye adsorbed. It is seen that three distinct curves result for the three coals.

No general law has yet been given that expresses the relation between the surface or weight of solid and the amount of adsorption. The expression known as Freundlich's isotherm, $x = kc^m$, where $x$ is the weight of dye adsorbed per gram of solid or per unit of surface, $c$ is the concentration at equilibrium, and $k$ and $m$ are constants, holds for many substances over a short range. It did not hold for the data shown in Tables I, II and III, although it did hold for a special test where the surface placed in the dye solution was varied 4 to 1 by variation in the weight of coal of a given size.

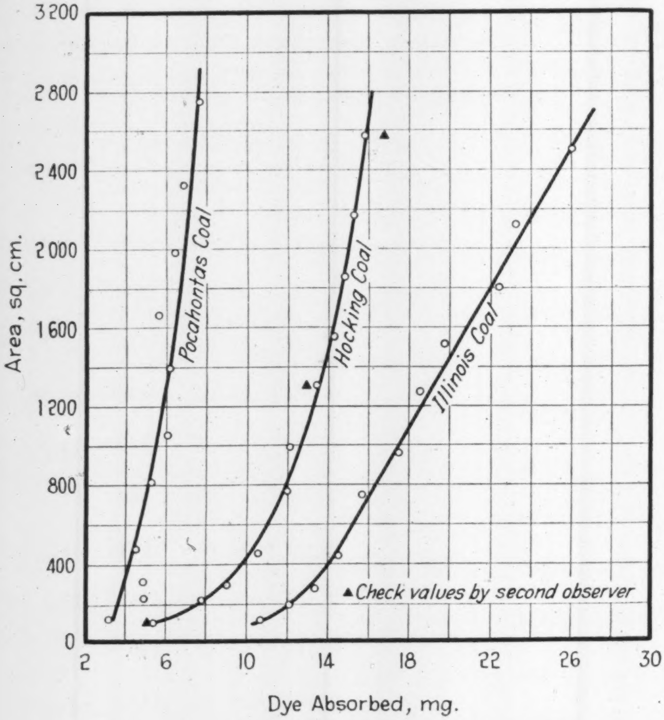

FIG. 1.—Relation of Dye Adsorbed to Area of Coal.

By trial it was found that the data on the Hocking coal, which were obtained first, would plot as a straight line following either the equation $s = be^{mx}$ or $x = a - \frac{a}{s^k}$, where $s$ is the surface in square centimeters, $x$ is the grams of dye adsorbed, $a$ is the original amount of dye, $e$ is the base of natural logarithms, and $b$, $m$, and $k$ are constants. The first equation is not logical as it states that if $x$, the dye adsorbed, is zero, $s = b$; that is, a certain amount of surface would adsorb no dye. The second equation is more nearly correct, for, as $s$ approaches infinity, $x$ approaches $a$; although, as $x$ approaches zero, $s$ does not approach zero, the data held closely to the curve over the range covered.

Figure 2 shows the data for the three coals plotted according to the second equation expressed as $\log \dfrac{a}{a-x} = k \log s$. Over the range of surface area covered with both the Hocking and Pocahontas coals, the data fall very close to straight lines; up to approximately 100-mesh coal the data for Illinois coal also follow a straight line, but depart widely for the smaller sizes.

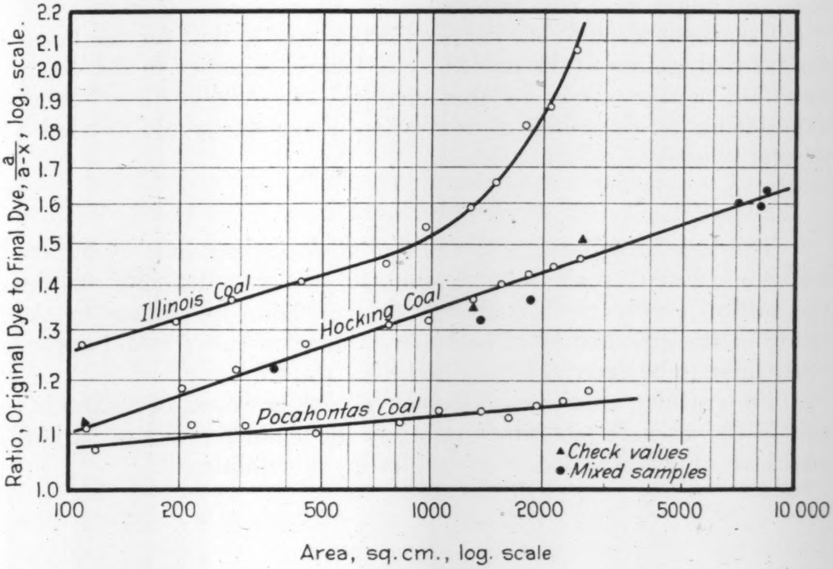

FIG. 2.—Relation of Area to Ratio of Original Dye to Remaining Dye.

The result found—that the adsorption of a given calculated surface is not the same for the three coals—is not particularly surprising, although it had been hoped that they would be found to be the same. The amount of dye adsorbed by the three coals falls in the same order as their inherent moisture or as the moisture they would take up from the atmosphere. This is further confirmation of the fact that the adsorption of dye is not limited to the external surface of the particles.

The sudden increase in the adsorption of Illinois coal finer than 100-mesh is not explained other than by the assumption that crushing to this size opens much more internal surface.

The tendency of the finer coals not to wet, but to remain on the surface of the solution or on the sides of the flasks, caused some difficulty. The use of a small amount of saponin to produce wetting was tried with no success. The use of alcohol or alcohol-water solutions might have removed this difficulty.

The slight slope to the curves, particularly to that for the Pocahontas coal, points to the possibility of lack of accuracy in the determination of the surface by this method. It may be that further experiments on the initial concentration of the dye would develop a greater sensitivity for the method.

## CONCLUSIONS

The lack of agreement in the dye adsorbed per unit of surface for the different coals renders doubtful the practicability of the application of the method to the determination of surface in the study of the grindability of coal. A calibration curve would apparently have to be determined for each coal studied.

For crushing studies on one type of coal, however, or as another tool in the research laboratory to study the characteristics of various coal beds, dye adsorption offers interesting possibilities.

# DISCUSSION

Mr. H. H. Lowry[1] (*presented in written form*).—The paper by Sherman, Irion and Rogers is of considerable interest in view of the fact that the authors have attempted to use a method, which was developed for characterizing "active carbons," for determining the external surface of pulverized coals. I wish particularly to support their conclusion that the adsorption of dye is not limited to the external surface of the particles.

Paneth and Radu[2] cited experimental evidence to show that the adsorption of 1 mg. of methylene blue by diamond, a crystalline form of carbon, corresponds to 1 sq. m. of surface. This figure has been taken over for other forms of carbon by several workers[3] who have studied the methylene blue method for determination of surface. Applying the figure of 1 sq. m. per mg. adsorbed to the data of Table III on the adsorption of methylene blue by various sieve fractions of Illinois coal, it can be readily calculated that the "total surface" given, ranges only from 0.1 to 1.0 per cent of the "methylene blue surface." In other words, the external surface contributes at the maximum only 1 per cent of the surface effective in the adsorption of the dye. Since only the external surface produced by pulverizing is of interest for determining the "grindability" of coal, it can be concluded that adsorption methods are inherently not adapted for this determination.

Mr. P. S. Roller[4] (*presented in written form*).—In this study of the adsorption of methylene blue by powdered coal, one is impressed by the enormous adsorption observed. For example, for the coarsest fraction of Hocking coal, the adsorption assuming a cubical dye molecule, is over 400 times that corresponding to a monomolecular layer. Additional surface due to superficial cracks and roughness usually involves a factor of 2 or 3 times the apparent surface. It would appear then that the methylene blue molecule has penetrated by diffusion or solution into the coal structure and become fixed by interior adsorption or chemical reaction. Further testimony for a dye penetration effect is found in the small increase in adsorption for a relatively great increase in superficial surface.

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[1] Director, Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] *Berichte* der deutschen chemischen Gesellschaft, Vol. 57, p. 1221 (1924).

[3] For instance see E. Berl and W. Herbert, "Zur Wertbestimmung aktiven Kohlen," *Zeitschrift für angewandte Chemie*, Vol. 43, p. 904 (1930); also H. Burkhardt, "Über die Herstellung von aktiven Kohlen," Dissertation Darmstadt (1927).

[4] Associate Chemist, Nonmetallic Minerals Experiment Station, U. S. Bureau of Mines, New Brunswick, N. J.

The results observed conceivably might be clouded by experimental difficulties not discussed in the paper. Attention is directed in this connection to the question of the purity of the dye. Methylene blue in solution is decomposed by light and by atmospheric exposure. It has been found further[1] that the ordinary dye will show an adsorption effect when the purified recrystallized dye will not.

The non-proportionality of the amount of dye adsorbed to the surface presented by different particle sizes means that the adsorption is a function not only of the surface but also of the particle size distribution. The total surface represented by highly active unsaturated components in the coal may also vary with the degree of grinding. This may account in part for the results with Illinois coal. Finally, the adsorption of methylene blue by coal apparently depends on the degree of freshness of the pulverized coal.[2]

The authors state that the air analyzer described by Roller is similar to but more refined than that of Bouton and Pratt. This is hardly the case. The apparatus of Roller departs in a fairly radical manner from analyzers previously described. This is so chiefly in the sense that agitation of the powder charge is considered detrimental and is avoided just as much as quiescence of the charge. Instead of either of these conditions, the charge is made to experience a uniform motion combining translation and rotation that is desirable from a theoretical point of view and practically is found to be unusually effective.

Actually the air analyzer has been used in some preliminary work with coal dust and a carbon black with results as to speed and accuracy that appear to approach those previously obtained with portland cement.[3] For the carbon black the first cut was made at 3 microns, and for the relatively coarse coal dust at 5 microns.

MR. R. A. SHERMAN.[4]—I should like to say that Mr. Roller has closely followed our work and the conclusions given in his discussion are by no means hindsight; they are foresight, because he prophesied this when we started in with the work. Being a physical chemist he was quite sure that the dye would not be adsorbed in a monomolecular layer but we did not care whether or not it was adsorbed in a monomolecular layer if we could get some relation between the surface as calculated and the dye adsorbed so that we could reasonably accurately calculate the area of an unknown sample from the dye adsorbed.

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[1] Teriwellen, "Adsorption," *Zeitschrift für physikalische Chemie*, Vol. 153 A, p. 52 (1931).

[2] Pentegov and Nyankovskii, "The Adsorption Characteristics of Coal," *Chemical Abstracts*, Vol. 25, p. 5193 (1932).

[3] P. S. Roller, "Measurement of Particle Size with an Accurate Air Analyzer: The Fineness and Particle Size Distribution of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 607 (1932).

[4] Fuel Engineer, Battelle Memorial Institute, Columbus, Ohio.

# AGGLUTINATING-VALUE TEST FOR COAL[1]

By W. A. Selvig,[2] B. B. Beattie[3] and J. B. Clelland[4]

## Synopsis

The agglutinating-value test is a small-scale laboratory test giving information of value regarding the coking and caking properties of coal. It is an approximate measure of the quantity and quality of that material in coal which fuses and becomes plastic on heating. This property of agglutination is necessary for coals used for coke manufacture but often causes trouble in burning coals on grates due to obstruction of air through the fuel bed.

Various factors affecting the method of test were investigated and a recommended procedure is given whereby 20-g. mixtures of coal ground to pass a No. 200 sieve and closely sized Ottawa sand are carbonized for 20 minutes at a temperature of 950 C. in the standard furnace used for determination of volatile matter in coal.

The agglutinating values are expressed in terms of the crushing strength in kilograms of these carbonized buttons. For routine testing it is recommended that buttons be made at the following ratios of sand to coal: 15:1, 20:1, 25:1, and 30:1. The crushing strengths of these carbonized buttons are plotted against the ratios of sand to coal. Such curves give considerable information regarding the agglutinating properties of various coals.

Coking coals when heated progressively in the absence of air fuse and become plastic. Some coals under these conditions do not fuse and are therefore not suitable for manufacture of coke. Between these coking and non-coking coals are poorly coking coals which possess in a varying degree the property of fusing when heated.

As early as 1870, Richters[5] proposed a laboratory method of testing coal for its agglutinating, or coking, properties. He mixed varying amounts of powdered quartz with coal and carbonized the mixture in a platinum crucible over a Bunsen burner. The agglutinating value taken was the ratio of quartz to coal which gave a coke button that would just sustain a 500-g. weight. There is a lack of agreement among coal chemists as to the value of such small-scale laboratory tests to evaluate coals for coke-making. The strength of commercial coke is usually measured by means of the well-known shatter[1] and tumbler tests.[2] It is probably asking too much to expect a laboratory agglutinating-value test to correlate closely with the strength of commercial coke, which is affected to a considerable degree by the heat treatment to which the coal is subjected in commercial coking processes.

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[1] Published by permission of the Director, U. S. Bureau of Mines, the Carnegie Institute of Technology, and the Mining Advisory Board.

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[3] Research Fellow, Carnegie Institute of Technology, Pittsburgh, Pa., 1930–31.

[4] Research Fellow, Carnegie Institute of Technology, Pittsburgh, Pa., 1931–32.

[5] E. Richters, "Einfaches Verfahren zur Vergleichenden Bestimmung der Backfähigkeit der Steinkohlen," *Dingler's Polytechnischer Journal*, Vol. 195, p. 71 (1870).

The test has been shown to be of value in detecting deterioration of coking properties of coal due to storage.[3,4] It should also give information of value as to the extent to which various coals may cake when burned on grates. Such caking may partly obstruct the flow of air through the fuel bed and influence the burning characteristics of the coal. When burning small sizes of coal, caking is generally desirable as it converts the small fuel into larger pieces of coke and consequently a lower draft is required than for the same depth of fuel fed with a non-caking coal. Another probable use of the test is in connection with coals for use in small domestic stokers. For this purpose coals which cake excessively cause trouble in the operation of these stokers. According to Fulweiler and Cleveland[5] it may be of value for selecting coals for water gas generators, as for this purpose coals which fuse excessively are undesirable.

Marshall and Bird[6] reviewed and summarized the various methods recommended by previous investigators and proposed a method of test incorporating what they believed to be the desirable features of these methods. The method adopted consists of mixing the finely ground coal with ten parts by weight of sized Ottawa sand, the mixture compacted by pressure in a porcelain crucible, and heated for 20 minutes at a temperature of 950 C. in an electrically heated furnace. The resulting carbonized button is then crushed in a compression machine and the crushing strength, expressed in kilograms, is taken as the agglutinating value. Johnson and Yancey[7] made a study of various factors affecting the method of test as proposed by Marshall and Bird. Their investigation clearly showed the need of careful standardization of the test to obtain comparable results in different laboratories.

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[1] Standard Method of Shatter Test for Coke (D 141 – 23), 1930 Book of A.S.T.M. Standards, Part II, p. 738.

[2] Standard Method of Tumbler Test for Coke (D 294 – 29), 1930 Book of A.S.T.M. Standards, Part II, p. 750.

[3] S. M. Marshall, H. F. Yancey and A. C. Richardson, "Loss in Agglutinating Power of Coal Due to Exposure," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Coal Division, p. 389 (1930).

[4] H. J. Rose and J. J. S. Sebastian, "Changes in Properties of Coking Coals Due to Moderate Oxidation During Storage," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Coal Division, p. 556 (1930).

[5] W. H. Fulweiler and T. K. Cleveland, "The Agglutinating Value of American Coals," *Proceedings*, Am. Gas Assn., p. 1399 (1927).

[6] S. M. Marshall and B. M. Bird, "Test for Measuring the Agglutinating Power of Coal," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Coal Division, p. 340 (1930).

[7] K. A. Johnson and H. F. Yancey, "A Study of the Marshall-Bird Test for Determining the Agglutinating Value of Coal," U. S. Bureau of Mines *Report of Investigations 3011* (1930).

## SCOPE OF INVESTIGATION

The purpose of the investigation described herein was to continue the study of the variables affecting the method with a view of standardizing the method of test. The work was conducted at the Pittsburgh Experiment Station of the U. S. Bureau of Mines during the period August, 1930, to June, 1932, with the aid of Fellows of the Carnegie Institute of Technology, supported by funds from the National Coal Association.

The source of the coals shown in the various tables is designated as to state and bed only. A comparison of the agglutinating value for a given coal bed, as given in the tables, may show differences, as the samples of coal were not always confined to the same mine. Even in case of coal from the same mine some changes in agglutinating value during the time interval between the various tests may have occurred due to partial oxidation of the coal on storage in the laboratory. The results for a given bed as shown in the individual tables were, however, obtained from coal from the same mine and from tests made at the same time.

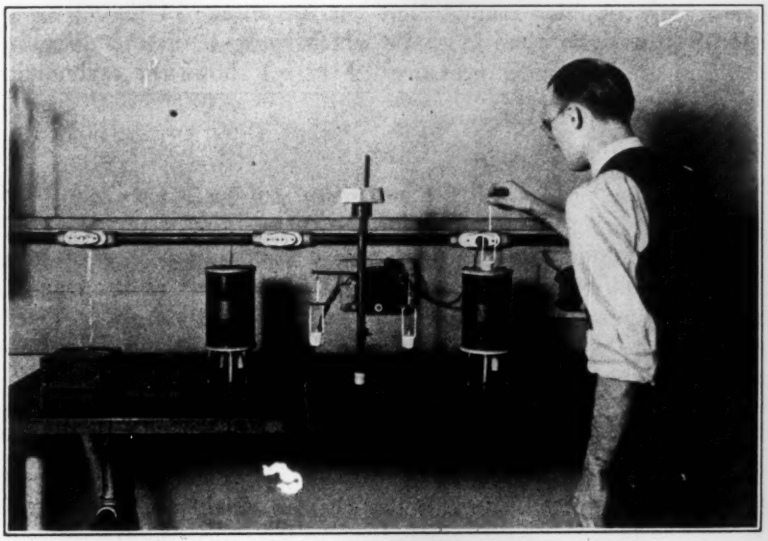

FIG. 1.—Carbonizing Furnaces and Accessories.

The electric tube furnace regularly employed in this country in the standard method for determination of volatile matter in coal[1] was found to be well adapted for this test. The advantage of using this furnace is that it is standard equipment found in most laboratories making coal analyses. With this furnace only one crucible containing the sand-coal mixture can be used at a time. One such furnace is sufficient if only a limited number of coals need be tested; otherwise two of these furnaces will suffice.

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[1] Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 30), 1930 Book of A.S.T.M. Standards, Part II, p. 689.

The crucible selected for carbonizing the sand-coal mixture was the Coors cylindrical porcelain crucible No. 390 (inside diameter, 28 mm.; inside height, 30 mm.; capacity, 18 ml.). This crucible has a capacity of 20 g. of the mixture of coal and sand, which amount was used in the tests described herein. The crucible is provided with a porcelain cover. This crucible has the advantage of giving a carbonized button cylindrical in shape which shows a distinct breaking point when crushed under pressure. Figure 1 shows the carbonizing furnaces and accessories.

## TIME OF CARBONIZATION

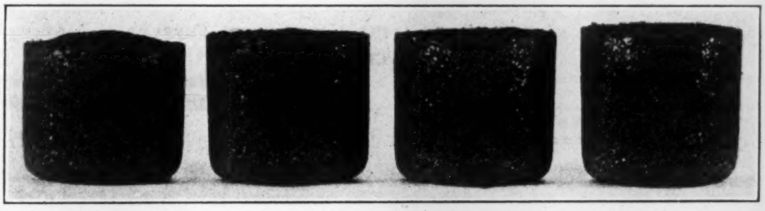
On introducing the crucible with the sand-coal mixture into the standard volatile-matter furnace, regulated to a temperature of 950 C., the furnace temperature drops 40 to 80 C. in about one minute; it then gradually rises, and with a slight adjustment of the rheostat reaches a temperature of 950 C. in the crucible zone about fifteen minutes after the charge is inserted. A 20-minute carbonization period was adopted so as to insure complete carbonization at 950 C.

## CALIBRATION OF FURNACE

A thermocouple is installed permanently in the volatile-matter furnace. It is inserted through the bottom of the furnace so that the hot junction is just below the bottom of the crucible. To calibrate the furnace one of the porcelain crucibles is filled with sand and placed in the furnace. A thermocouple is inserted into the sand so that the end of the protection tube is in contact with the bottom of the crucible. The furnace rheostat is then adjusted until a constant temperature of 950 C. is registered by the thermocouple in the crucible. The temperature of the fixed thermocouple of the furnace is then recorded, and this temperature corresponds to 950 C. in the crucible zone when a crucible is in the furnace. The stirrup and crucible are then removed and it will be noted that the fixed furnace thermocouple will then register a drop in temperature of approximately 30 C. This drop in temperature of the furnace couple is due to radiation from the end of the thermocouple protection tube to the cold end of the furnace tube. The temperature registered by the furnace couple is recorded, and this temperature corresponds to 950 C. in the crucible zone when there is no crucible in the furnace.

### INERT MATERIAL

Closely sized Ottawa sand with well-rounded grains was accepted as the most practical inert material for mixing with the coal. Such sand has an advantage over other materials that have been proposed, such as electrode carbon, coke, and anthracite, because of its uniformity of surface and shape. The objection raised by some investigators that sand might fuse with the ash-forming mineral constituents of coal appears to have no foundation. Microscopic examination of the sand grains from carbonized buttons of various coals showed no indication of any slagging action. Mixtures of calcite and sand were heated under the conditions of the agglutinating-value test and no indication of any reaction between the calcite and sand could be detected.



No glycerine

1 drop of glycerine

2 drops of glycerine

3 drops of glycerine

FIG. 2.—Effect of Varying Amounts of Glycerine on Buttons of Pittsburgh Bed Coal.

The sand is carefully sized in the laboratory and that portion passing through a No. 45 sieve (350-micron opening) and remaining on a No. 60 sieve (250-micron opening) is retained for the agglutinating-value test. Cleaning of the sand was first accomplished by washing it with carbon tetrachloride; later it was found that the sand could be cleaned more effectively by washing it with water, followed by boiling with dilute hydrochloric acid.

### PREPARATION OF SAND-COAL BUTTONS

Care must be used in mixing the sand and coal to prevent segregation. Marshall and Bird found that the best method of preventing such segregation was first to moisten the sand with a small amount of glycerine, then to add the coal, and stir the mixture thoroughly with a spatula. The use of water was found undesirable due to the varying amount of evaporation during the mixing process. Johnson and Yancey prepared mixtures of sand and coal using 1, 2 and 3 drops of glycerine, respectively. They found that the amount of glycerine used was an important factor, as the agglutinating values decreased with increase in the amount of glycerine.

To determine the cause of variation of agglutinating value with different amounts of glycerine, tests were made on two coals. The air-dry coal was ground to pass a No. 60 sieve and mixed with 10 parts by weight of sand; the total weight of sand and coal was 20 g. For one mixture no glycerine was used, for the others the glycerine varied from 1 to 3 drops. Each drop of glycerine weighed 0.07 g. The mixtures were transferred to the carbonizing crucibles and compacted for 30 seconds, under a pressure of 3½ kg. Examination of these compacted buttons showed that the glycerine affected the volume occupied by the sand-coal mixtures, as evidenced by differences in the height of the buttons. The button made with one drop of glycerine was higher by 1.4 mm. than the button made with no glycerine. The button with 2 drops of glycerine had a height greater by 2.6 mm. than that with no glycerine, and the button with 3 drops of glycerine was 2.9 mm. higher.

TABLE I.—VARIATION OF AGGLUTINATING VALUE WITH DIFFERENT AMOUNTS OF GLYCERINE.

| COAL | AMOUNT OF GLYCERINE, DROPS | AGGLUTINATING VALUE, KG. |
|---|---|---|
| Elkhorn bed, Kentucky | 0 | 9.2 |
| | 1 | 7.3 |
| | 2 | 6.0 |
| | 3 | 5.0 |
| Pittsburgh bed, Pennsylvania | 0 | 7.7 |
| | 1 | 7.8 |
| | 2 | 7.8 |
| | 3 | 7.5 |

After carbonizing the buttons for 20 minutes at 950 C. it was noted that the carbonized buttons varied in height. Figure 2 shows the variation in height of the carbonized buttons of Pittsburgh bed coal. Table I gives the agglutinating values for two coals. The table shows that varying the amount of glycerine has a pronounced effect on the agglutinating value of the Elkhorn bed coal, but not with the Pittsburgh bed coal. The agglutinating values given in the various tables of this paper are averages of five or six individual determinations calculated according to the procedure given under the proposed method of test at the end of the paper.

Examination of Fig. 2 shows that the Pittsburgh bed coal had an excess of material which fused on heating, as evidenced by the convex top of the button made without the use of glycerine. The top of the corresponding button for the Elkhorn bed coal was slightly concave, showing that there was insufficient agglutinating material to fill the spaces between the sand grains. For some coals, especially those with relatively small amounts of agglutinating material, the amount of glycerine used will materially affect the agglutinating values. Increasing the amount of glycerine increases the volume occupied by the sand-coal mixture, with consequent increase in the voids between the sand grains. One drop of glycerine weighing 0.07 g. was used in all subsequent tests. This was conveniently obtained by the use of a suitable burette.

TABLE II.—EFFECT OF FINENESS OF COAL ON AGGLUTINATING VALUE.

| Coal | | Ratio, Sand to Coal | Agglutinating Value, kg. | | | |
|---|---|---|---|---|---|---|
| State | Bed | | Coal Passing a No. 60 Sieve (250 micron) | Coal Passing a No. 100 Sieve (149 micron) | Coal Passing a No. 200 Sieve (74 micron) | Coal Passing a No. 325 Sieve (44 micron) |
| British Columbia | | 10:1 | 9.5 | 10.0 | 12.7 | 14.9 |
| | | 14:1 | 5.9 | 6.4 | 7.4 | 6.9 |
| Pennsylvania | Pittsburgh | 10:1 | 9.0[a] | 9.7[a] | 10.7[a] | 13.4[a] |
| | | 14:1 | 6.8 | 7.7 | 8.7 | 8.8 |
| Maryland | Davis | 10:1 | 12.3[a] | 15.3[a] | 16.5[a] | 15.2[a] |
| | | 14:1 | 8.8 | 8.8 | 9.2 | 8.6 |
| West Virginia | Chilton | 10:1 | 6.1 | 6.9 | 7.4 | 7.5 |
| | | 14:1 | 4.1 | 4.4 | 4.9 | 6.0 |
| West Virginia | No. 2 Gas | 10:1 | 4.3 | 5.6 | 6.8 | 6.9 |
| | | 14:1 | 3.0 | 3.7 | 4.6 | 5.0 |
| Kentucky | Elkhorn | 10:1 | 6.0 | 7.8 | 8.7 | 8.3 |

[a] Abnormal button showing cavity inside.

## FINENESS OF COAL

A number of coals were selected to test the effect of varying the fineness of the coal used to make the sand-coal buttons. Samples of air-dry coal were ground to just pass a No. 60 sieve. Portions of this coal were ground further to just pass Nos. 100, 200 and 325 sieves. Agglutinating values were then determined at the 10:1 and 14:1 ratios of sand to coal. Table II gives the results of these tests. The agglutinating values generally increase with increase of fineness of the coal up to and including the coal crushed to pass a No. 200 sieve. Grinding the coal to pass a No. 325 sieve gave for some coals

TABLE III.—EFFECT OF TEMPERATURE OF CARBONIZATION ON AGGLUTINATING VALUE.

| Coal | | Carbonization Temperature, deg. Cent. | Agglutinating Value, kg. | | | | |
|---|---|---|---|---|---|---|---|
| State | Bed | | Ratio of Sand to Coal, 8:1 | Ratio of Sand to Coal, 10:1 | Ratio of Sand to Coal, 12:1 | Ratio of Sand to Coal, 14:1 | Ratio of Sand to Coal, 16:1 |
| West Virginia | Pocahontas 4 | 800 | 24.0 | 15.5 | 11.6 | 7.5 | 4.3 |
| | | 950 | 12.6 | 8.3 | 5.5 | 3.4 | 2.2 |
| Pennsylvania | Pittsburgh | 800 | 10.3[a] | 11.0 | 9.3 | 8.0 | 6.9 |
| | | 950 | 5.7[a] | 7.8 | 7.4 | 5.7 | 4.5 |
| Maryland | Davis | 800 | 13.4[a] | 16.1 | 12.0 | 9.9 | 8.0 |
| | | 950 | 8.2[a] | 11.9 | 10.5 | 8.8 | 6.7 |
| Pennsylvania | Pittsburgh[b] | 800 | [c] | 8.0[a] | 9.4[a] | 9.4 | 8.7 |
| | | 950 | 7.9[a] | 7.0[a] | 7.9[a] | 7.3 | 6.2 |
| Alabama | Mary Lee | 800 | 7.9[a] | 12.0[a] | 11.7 | 10.1 | 8.8 |
| | | 950 | 7.5[a] | 9.9[a] | 9.9 | 8.6 | 7.1 |

[a] Abnormal button showing cavity inside.
[b] Connellsville coking coal.
[c] Not determined.

TABLE IV.—RELATIONSHIP OF AGGLUTINATING VALUE TO SIZE OF SAND.

| Coal | | Ratio, Sand to Coal | Agglutinating Value, kg. | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Uncrushed Sand | | | Crushed Sand | | | |
| State | Bed | | Passing a No. 35 Sieve and Retained on a No. 45 Sieve | Passing a No. 45 Sieve and Retained on a No. 60 Sieve | Passing a No. 60 Sieve and Retained on a No. 100 Sieve | Passing a No. 60 Sieve and Retained on a No. 100 Sieve | Passing a No. 100 Sieve and Retained on a No. 140 Sieve | Passing a No. 140 Sieve and Retained on a No. 200 Sieve | Passing a No. 200 Sieve and Retained on a No. 325 Sieve |
| Pennsylvania | Pittsburgh | 10:1 | 6.3 | 7.4 | 8.6 | 5.3 | 3.6[a] | 2.1[a] | 2.4[a] |
| | | 14:1 | 4.4 | 5.9 | 5.6 | 3.4 | 2.5 | 2.7 | 2.0[a] |
| Virginia | Taggart | 10:1 | 6.5 | 8.7 | 10.5[a] | [b] | 5.3[a] | 4.5[a] | 3.1[a] |
| | | 14:1 | 6.0 | 8.9 | 9.9 | [b] | 4.1 | 3.4 | [b] |

[a] Abnormal button showing cavity inside.
[b] Not determined.

TABLE V.—EFFECT OF FINELY GROUND INERTS ON AGGLUTINATING VALUE.

| Coal | | Inert Material | Agglutinating Value, kg. | | | | | |
|---|---|---|---|---|---|---|---|---|
| State | Bed | | No Inert Material in Coal Mixture | 10 per cent Inert Material in Coal Mixture | 20 per cent Inert Material in Coal Mixture | 30 per cent Inert Material in Coal Mixture | 40 per cent Inert Material in Coal Mixture | 50 per cent Inert Material in Coal Mixture |
| West Virginia | Chilton | calcite | 7.4 | 10.3 | 14.3 | 15.9 | 16.0 | 10.5[a] |
| Pennsylvania | Pittsburgh | calcite | 8.2 | 12.4 | 16.2 | 21.6 | 20.0 | 14.6[a] |
| | | sand | 8.2 | 10.7 | 14.0 | 16.8 | 18.6 | 10.7[a] |

[a] Abnormal button showing cavity inside.

higher values than the coal ground to pass the No. 200 sieve; for some coals the agglutinating values of the coal ground to pass the No. 325 sieve were less than those which passed the No. 200 sieve. The authors believe that the most consistent results will be obtained by grinding the coal to pass a No. 200 sieve. Grinding finer than this is not practical.

## EFFECT OF TEMPERATURE OF CARBONIZATION

Johnson and Yancey, using the procedure recommended by Marshall and Bird, determined agglutinating value of five coals at temperatures of 750, 850, 950 and 1050 C. They found that the agglutinating value of all the coals tested decreased with increase of temperature of carbonization up to 950 C. For four of the five coals the values obtained at 1050 C. were somewhat higher than at 950 C.; for one coal the value at 1050 C. was slightly lower. They concluded from these tests that a carbonizing temperature of 950 C. gives the minimum agglutinating value and that this temperature is satisfactory for the test.

Tests were made by the authors with a number of coals using carbonizing temperatures of 800 and 950 C. and buttons prepared with different amounts of sand. Table III shows the results of these tests. The agglutinating values at 950 C. are in all cases lower than those obtained at 800 C.

## EFFECT OF SIZE OF SAND

The sand used in the tests previously described was sized to pass a No. 45 sieve and remain on a No. 60 sieve. Other sizes of sand were tried, namely, that passing a No. 32 sieve and retained on a No. 45 sieve, and that passing a No. 60 sieve and retained on a No. 100 sieve. Also some of the sand was crushed and sieved into various sizes by means of sieves Nos. 60, 100, 140, 200 and 325.[1] Buttons weighing 20 g. were made with two coals at ratios of 10 parts sand to 1 part coal, and 14 parts sand to 1 part coal. The coal samples were crushed to pass a No. 200 sieve. Table IV shows the results of these tests. Increasing the fineness of the uncrushed sand increased the agglutinating value. The results obtained with the crushed sand are considerably lower than with the uncrushed sand.

Inspection of the carbonized buttons prepared with the uncrushed sand showed no detectable difference in volume for the different sizes of sand. The carbonized buttons made with crushed sand showed an increase of volume with fineness of sand. The carbonized button prepared with the crushed sand passing a No. 60 sieve and retained on a No. 100 sieve occupied a larger volume than the carbonized button prepared with the corresponding size of uncrushed sand. These tests show that the size of sand and the shape of the grains are important factors in the method of test.

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[1] For specifications concerning sieve openings see Standard Specifications for Sieves for Testing Purposes (E 11 - 26), 1930 Book of A.S.T.M. Standards, Part II, p. 1119.

## EFFECT OF FINE INERTS

Calcite and sand were ground to pass a No. 200 sieve and mixed in varying amounts with coal crushed to the same fineness. Buttons weighing 20 g. were made with these mixtures and with sand passing a No. 45 sieve and retained on a No. 60 sieve. The amount of coarse sand was corrected for the amount of finely ground inert matter that had been added to the coal. In this way each button had the same amount of coal, and the ratio of inerts (finely ground inert plus coarse sand) was maintained at 10 parts to 1 part of coal. The buttons were carbonized at 950 C. for 20 minutes.

TABLE VI.—EFFECT OF METHOD OF CLEANING SAND ON AGGLUTINATING VALUE.

| Coal | | Ratio, Sand to Coal | Agglutinating Value, kg. | |
|---|---|---|---|---|
| **State** | **Bed** | | Sand Cleaned with Carbon Tetrachloride | Sand Cleaned with Water and HCl (1:1) |
| Pennsylvania | Pittsburgh | 10:1 | 10.5 | 7.9 |
| West Virginia | Alma | 10:1 | 9.0 | 6.9 |
| | | 12:1 | 7.4 | 6.2 |
| West Virginia | Pocahontas 4 | 10:1 | 11.5 | 11.7 |
| | | 12:1 | 8.2 | 7.3 |
| Alabama | Pratt (washed) | 12:1 | 10.1 | 8.0 |
| Alabama | Pratt (unwashed) | 12:1 | 11.2 | 8.8 |
| Pennsylvania | Pittsburgh (Connellsville) | 14:1 | 10.2 | 8.8 |
| | | 16:1 | 8.6 | 7.4 |

Table V gives the results of these experiments. Finely ground sand and calcite increased the agglutinating value up to the mixtures containing 40 per cent of fine inerts. The mixtures containing 50 per cent of fine inerts show a decrease in agglutinating value. These buttons, however, were abnormal in structure as they showed cavities when sectioned. These cavities weakened the buttons. It is probable that the increase of agglutinating value with addition of fine inerts is caused in part at least by the resulting decrease in spaces between the coarse sand grains as well as to the increase in surface of the inert material. For one coal both sand and calcite were used and in this case the calcite increased the agglutinating values more than did the sand. Careful examination of the crushed buttons under the microscope did not show any indications of fusion between the calcite and the sand.

## METHOD OF CLEANING SAND

The sized sand used in the tests previously described was cleaned by washing with carbon tetrachloride. Shaking the sand with this liquid was found to be unsatisfactory for removal of the small amount of clay material associated with the sand. Tests made by shaking the sand with water showed that a considerable portion of this finely divided clay could be removed.

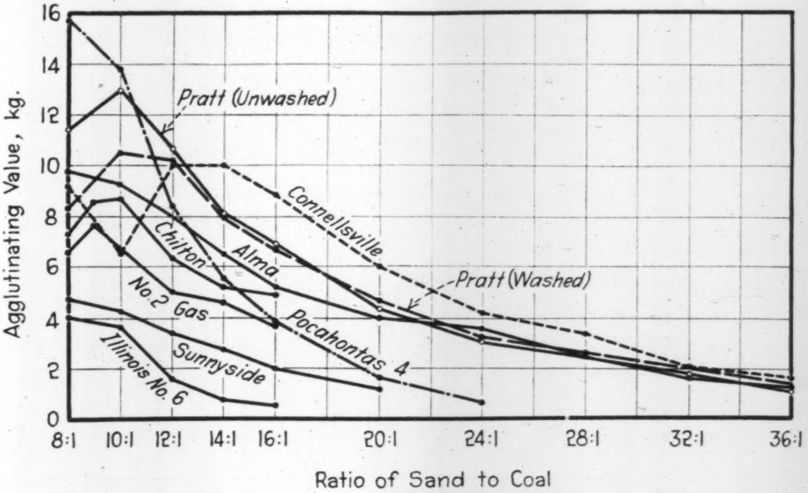

FIG. 3.—Agglutinating Values at Different Ratios of Sand to Coal.

A satisfactory method of cleaning the sand is to shake it vigorously with water, decant the suspended matter, repeat until practically no more clay is removed, then boil the sand for one-half hour in dilute hydrochloric acid (1:1), filter and wash free of chlorides and dry thoroughly. Sand prepared in this manner is practically free of clay material and surface coatings.

Agglutinating-value tests were made on a number of coals with sand cleaned by carbon tetrachloride alone, and with sand cleaned by treating with water and dilute hydrochloric acid. The coal was ground to pass a No. 200 sieve. Table VI shows the results of these tests. Consistently lower results were obtained with sand cleaned with water and hydrochloric acid, which method gave a cleaner sand than could be obtained with carbon tetrachloride.

## DETERIORATION OF SAMPLES BY OXIDATION

It is advisable to use freshly mined coal for the agglutinating-value test. After pulverization in the laboratory the sample should be kept in stoppered containers to retard oxidation. Some coals, especially those which have relatively low agglutinating values, must not be kept for any length of time before testing.

A sample of Utah coal (Sunnyside bed) which showed an agglutinating value of 2.7 kg. at the 14:1 ratio of sand to coal had an agglutinating value of 1.7 kg. at the end of 14 days. This sample was pulverized to pass a No. 200 sieve and was stored in a tightly stoppered bottle. A sample of Illinois coal (No. 6 bed) crushed to pass a No. 60 sieve showed an agglutinating value of 3.7 kg. at the 10:1 ratio of sand to coal; after standing for 35 days in a tightly stoppered bottle the agglutinating value was only 0.3 kg. Tests made on a number of Appalachian coals showed no deterioration in agglutinating values of the coal pulverized to pass a No. 200 sieve after standing 14 days in tightly stoppered bottles.

TABLE VII.—PROXIMATE ANALYSIS OF COALS SHOWN IN FIG. 3, AIR-DRY BASIS.

| Coal | | | Moisture, per cent | Volatile Matter, per cent | Fixed Carbon, per cent | Ash, per cent | Agglutinating-Value Test | |
|---|---|---|---|---|---|---|---|---|
| State | Bed | Laboratory Number | | | | | Fineness of Coal, Sieve Number | Method of Cleaning Sand |
| Alabama | Pratt (washed) | A79220 | 1.0 | 32.2 | 63.6 | 3.2 | 200 | $CCl_4$ |
| | Pratt (unwashed) | A78090 | 1.0 | 30.2 | 59.5 | 9.3 | 200 | $CCl_4$ |
| Illinois | No. 6 | A69265 | 7.9[a] | 32.1 | 47.7 | 12.3 | 60 | $CCl_4$ |
| Pennsylvania | Pittsburgh (Connellsville) | A67055 | 0.6 | 29.9 | 60.1 | 9.4 | 200 | $CCl_4$ |
| Utah | Lower Sunnyside | A80086 | 3.1 | 39.4 | 51.4 | 6.1 | 200 | HCl |
| West Virginia | Chilton | A73643 | 1.3 | 36.5 | 57.9 | 4.3 | 200 | $CCl_4$ |
| | No. 2 Gas | A74912 | 1.9 | 38.8 | 56.6 | 2.7 | 200 | $CCl_4$ |
| | Alma | A74396 | 1.4 | 39.1 | 53.8 | 5.7 | 200 | $CCl_4$ |
| | Pocahontas 4 | A66709 | 0.2 | 15.6 | 78.3 | 5.9 | 200 | $CCl_4$ |

[a] Sample not air-dried, analysis is on the as-received basis.

## SAND-COAL RATIOS

Tests were made on a number of coals by varying the ratio of sand to coal over a wide range. For some coals these ratios ranged from 8 parts of sand to 1 part of coal to 36 parts of sand to 1 part of coal. The results for some of these coals were plotted and are shown in Fig. 3. The proximate analyses of these coals are shown in Table VII. The curves show that a number of the coals gave increasing agglutinating values with decreasing ratios of sand to coal up to a maximum point, and then the agglutinating value decreased as the ratio of sand to coal was decreased.

As an illustration, consider the behavior of the Pratt (washed) coal shown in Fig. 3. A pronounced rise in agglutinating value occurs with decrease in the sand-coal ratio up to the 12:1 ratio; the agglutinating value at the 10:1 ratio is only slightly higher than that at the 12 : 1 ratio; and at the 8:1 ratio of sand to coal the agglutinating value drops sharply. Typical sections of carbonized buttons of this particular coal are shown in Fig. 4. Button No. 1, representing the 8:1 ratio of sand to coal, shows a large cavity near the top and a heterogeneous structure at the bottom. Button No. 2, representing the 10:1 ratio, also shows a heterogeneous internal structure due principally to the cavity near the top. Buttons Nos. 3 and 4, which represent the 12:1 and the 14:1 ratios, respectively, are homogeneous in structure. It is obvious that cavities and a heterogeneous internal structure weaken the buttons and cause them to crush at relatively low pressures. Such buttons are not considered as suitable test specimens and should not be used in comparisons of the agglutinating value of different coals. The lowering of the agglutinating value with decrease in the sand-coal ratio was always found to be due to cavities and heterogeneous structures inside the carbonized buttons.

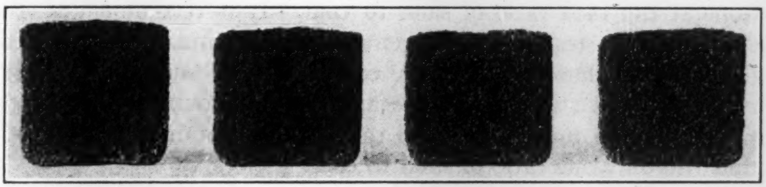
No. 1 No. 2 No. 3 No. 4

FIG. 4.—Internal Structure of Buttons of Pratt Bed (Washed) Coal.

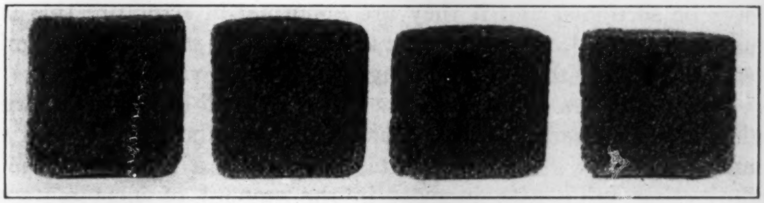
No. 1 No. 2 No. 3 No. 4

FIG. 5.—Internal Structure of Coal Buttons of Connellsville Coal.

The Pocahontas coal (Fig. 3) gives homogeneous buttons up to the 10:1 ratio of sand to coal; at the 8:1 ratio the buttons are slightly abnormal due to a small cavity near the top. The Connellsville coal, on the other hand, gives abnormal buttons at the 12:1 ratio. Figure 5 shows the internal structure of carbonized buttons of Connellsville coal at ratios lower than 14:1. Button No. 1 (6:1 ratio) shows a large cavity at the top and cavities at the bottom. Button No. 2 (8:1 ratio) shows a large cavity at the top and small cavities at the bottom. Button No. 3 (10:1 ratio) shows a large cavity at the top. Button No. 4 (12:1 ratio) shows a large cavity at the top. Buttons made at the 14:1 ratio of sand to coal were homogeneous in internal structure.

Most coals that have been tested have given homogeneous buttons at the 14:1 ratio of sand to coal. It is recommended that coals should be tested at different ratios of sand to coal and the agglutinating values plotted. A comparison of such curves gives considerably more information regarding the amount and quality of the agglutinating material in coal than can be obtained if the test is confined to only one ratio of sand to coal.

For a routine testing procedure it appears best to confine the agglutinating-value tests to the following ratios of sand to coal: 15:1, 20:1, 25:1, and 30:1. Buttons made at the 15:1 ratio should always be sectioned and if they are abnormal in structure this fact should be recorded. Curves made at these ratios should show significant differences in the agglutinating properties of various coals.

Figure 3 shows clearly that there is a marked difference between agglutinating properties of coals such as the Pocahontas coal and the Connellsville coal. The Pocahontas coal shows a rapid decrease in agglutinating value with increase in the amount of inert material. The Connellsville coal, on the other hand, has the property of binding together large amounts of inert material. One may well believe that the quality as well as the quantity of agglutinating material differs in various coals. Coals with low agglutinating values, such as the Illinois coal and the Utah coal, require careful attention to heat treatment in the preparation of commercial coke.

## PROPOSED METHOD OF TEST FOR AGGLUTINATING VALUE OF COAL

The following proposed method of test is based on the experimental work described in this paper. It is essential that the procedure outlined should be rigidly adhered to, as the test method is necessarily empirical in nature.

### APPARATUS

The following apparatus shall be used in the test:

*Furnace:*

A vertical electric-tube furnace as used in the standard method for volatile matter in coal shall be used. A suitable furnace available on the market is Hoskins type FA-120, Fieldner furnace. The furnace is provided with nickel-chromium stirrups; three of these should be obtained for each furnace. The furnace should be equipped with a rheostat for maintaining a temperature of 950 C. in the crucible zone.

*U. S. Standard Sieve Series Sieves:*

*For preparation of coal.*—Sieve No. 200 (74-micron opening).

*For preparation of sand.*—Sieve No. 45 (350-micron opening), and sieve No. 60 (250-micron opening).

*Porcelain Crucibles:*

*For mixing sand and coal.*—Coors high-form crucible size 2; 52 mm., top diameter; 25 mm., bottom diameter; 43 mm., height; capacity, 55 ml.

*For carbonizing buttons.*—Coors cylindrical crucible No. 390; 28 mm., inside diameter; 30 mm., inside height; capacity, 18 ml. Crucible to be provided with a porcelain cover, Coors No. 280, 35 mm., outside diameter. Two notches are ground in the sides of the cover where it comes in contact with the side wires of the crucible stirrup. The carbonizing crucibles should be selected stock, free from irregularities of shape.

*Compression Machine:*

Riehle compression-testing machine for agglutinating-value test equipped with constant-speed motor arranged for reversing. Dial marked in metric units, graduations in tenths, up to 25 kg. Rubber pads ⅜ in. thick should be cemented on the plates of the compression cage to equalize the pressure over any irregularities in the surfaces of the carbonized buttons. These pads are cut from the small end of a No. 12 rubber stopper.

*Button Compressor:*

For compressing sand-coal mixture in carbonizing crucible. See Fig. 1. This apparatus can be readily assembled in the laboratory. It should be weighted to give a pressure of 3500 g. on the sand-coal mixture. The lower end of the compressor should fit snugly, without binding, into the crucible so that the entire load of 3500 g. is applied to the mixture of sand and coal.

## SAND

For use as inert material. Ottawa sand for agglutinating-value test for coal shall pass a No. 40 sieve and be retained on a No. 60 sieve.

*Preparation of Sand:*

The Ottawa sand is carefully sized in the laboratory and that portion passing a No. 45 sieve and retained on a No. 60 sieve is kept for the test. This sized sand is placed in a large-mouth bottle, shaken vigorously with water, and the turbid water decanted: repeat this operation 5 times, then boil the sand with dilute hydrochloric acid (1:1) for ½ hr. Decant the acid, transfer the sand to a Buechner filter and wash with water until free of chlorides. Dry the sand thoroughly and transfer it to a covered container.

## PREPARATION OF COAL

The coal for the test is prepared by crushing air-dry coal to just pass a No. 200 sieve. If a ball mill is used for grinding, about 200 g. of air-dry, No. 20 coal is ground for 15 minutes. The coal is then put through a No. 200 sieve and the oversize crushed on a bucking board to just pass the sieve. If a ball mill is not available, the 20-mesh coal may be ground to just pass the No. 200 sieve by means of a bucking board. The grinding of the oversize and sieving should be done in successive stages. Excessive grinding should be avoided. The coal is kept in a well-stoppered container to retard deterioration by oxidation. The agglutinating-value tests should be made on freshly prepared samples.

## METHOD

Agglutinating-value tests are made at the following ratios of sand to coal: 15 parts sand, 1 part coal; 20 parts sand, 1 part coal; 25 parts sand, 1 part coal; and 30 parts sand, 1 part coal. The weight of each mixture is maintained constant and should be 20 g. The amount of sand and coal for individual determinations for these ratios is as follows:

| RATIO, SAND TO COAL | WEIGHT OF SAND, G. | WEIGHT OF COAL, G. |
|---|---|---|
| 15:1 | 18.750 | 1.250 |
| 20:1 | 19.048 | 0.952 |
| 25:1 | 19.231 | 0.769 |
| 30:1 | 19.355 | 0.645 |

The sand is weighed into a No. 2 Coors high-form crucible and moistened with one drop of glycerine. This drop of glycerine should weigh 0.07 g. A drop of this weight can be conveniently delivered from a suitable burette. The glycerine is mixed with the sand by stirring for one minute. To this damp sand is added the weighed portion of coal, after which the mixture is stirred for 2 minutes. It is important that these mixing operations be performed in a thorough manner. To do this the following procedure is followed for both mixing operations: The crucible, resting on a table, is turned with the left hand in a counter-clockwise direction. At the same time a small spatula, held in the right hand in nearly a vertical position, is drawn repeatedly from the bottom of the crucible towards the top and simultaneously is moved horizontally in a clockwise direction around the side of the crucible.

The mixture is carefully transferred into a Coors No. 390 cylindrical crucible and the top leveled by means of a square-end spatula. The mixture in the crucible is then compressed for 30 seconds under a weight of 3500 g. by means of the compression apparatus. After compression, the empty space left at the top of the crucible is filled with a loose blanket of carbonized coal and sand remaining from preceding tests, and the notched porcelain crucible cover placed on the crucible. The covered crucible is placed in the furnace stirrup and inserted into the furnace which has been regulated to a temperature of 950 C. ± 10 C. in the crucible zone. The calibration of the furnace has been already described. The crucible is left in the furnace for 20 minutes.

The furnace couple will record a temperature drop of from 40 to 80 C. in about one minute after the charge is inserted, depending on the position of the couple in relation to the crucible. The temperature will then gradually rise. The furnace rheostat is then adjusted so that a temperature corresponding to 950 C. ± 10 C. in the crucible zone is reached 15 minutes after the charge is inserted. This temperature should be maintained during the last 5 minutes of the carbonization period.

After carbonizing exactly 20 minutes the stirrup and crucible are removed from the furnace and the stirrup is suspended in the air until the crucible has cooled to a temperature at which it can be handled with one's fingers; it then is removed from the stirrup and allowed to cool to room temperature. The carbonized buttons cool to room temperature in approximately 1 hour. Pour off the blanket of loose carbonized material, but do not remove the carbonized button from the crucible. Invert the crucible over a piece of No. 00 sandpaper and gently rub the button to remove any irregularities on the top surface. Remove the button from the crucible and place it in the compression machine so that the sanded surface rests on the lower rubber pad. The button is carefully centered in the cage of the machine and then crushed. The crushing strength as registered on the dial of the machine is recorded to the nearest tenth of a kilogram.

Six buttons are carbonized and crushed for each of the ratios of sand to coal. An additional button is carbonized at the 15:1 ratio and is sectioned by cutting it in half, from top to bottom, by means of a spatula. This sectioned button is examined for the presence of cavities or other heterogeneous structure and if these occur the button is designated an abnormal button. Such abnormal buttons are not suitable test specimens for determination of crushing strength.

The average agglutinating value of each set of six buttons is calculated and the variations of the individual buttons from this average determined. If all six buttons are within 10 per cent of the average value, this value is accepted as the agglutinating value. If only one button shows a difference greater than 10 per cent of the average, the value for this button is discarded and the average of the remaining five buttons is taken as the agglutinating value. If more than one button shows variations in excess of 10 per cent of the average value the test is considered unsatisfactory, the results discarded, and another test made on six buttons.

It is recommended that the agglutinating values be plotted against the sand-coal ratios. A comparison of such curves for various coals will give considerably more information than can be derived from a comparison of agglutinating values at any one ratio of sand to coal.

# DISCUSSION

MR. A. C. FIELDNER[1] (*presented in written form*).—The standardization of agglutinating value tests for coal is of immediate importance in connection with the work of the Sectional Committee on Classification of Coals. It is apparent that the caking, or agglutinating, properties of coal are important criteria in placing the boundary lines between different classes of coal. For example, coals of highest rank, such as anthracite and semi-anthracite, are not agglutinating; likewise, the lowest-rank coals, namely, lignite and sub-bituminous coal, are not agglutinating. The agglutinating coals are all of bituminous rank, although not all bituminous coals agglutinate.

The agglutinating index is important, also, in the use classification of coal. Preliminary investigations have indicated that the plastic properties of bituminous coals are of great importance in fitting the proper type of mechanical stoker to different coals. It is believed that the determination of agglutinating index will, in the future, be equally important to the determination of the fusibility of coal ash. The one test gives an indication of the clinkering properties of a coal, while the other throws light on the caking properties in the fuel bed.

It is hoped that the test proposed by Mr. Selvig and his associates will be tried out by others and that it may serve as the basis for an A.S.T.M. standard test.

MR. O. O. MALLEIS[2] (*presented in written form*).—This paper describes a small-scale laboratory test giving information of value regarding the coking and caking properties of coals. A satisfactory laboratory test has been desired for a long time, as the results obtained from small-scale laboratory tests in the past usually have not been at all satisfactory. It is, therefore, to be hoped that the U. S. Bureau of Mines will be able to apply this agglutinating-value test to a large number of coals of which the coking properties are known in order to establish the merits of the test. An excellent start, of course, has already been made in its survey of the gas, coke, and by-product making properties of American coals.[3]

MR. F. E. LATHE.[4]—I am wondering whether this test could be modified to apply better to those coals which agglutinate only a little.

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[1] Chief Engineer, Experiment Stations Division, U. S. Bureau of Mines, Washington, D. C.

[2] Chemist, Pittsburgh, Pa.

[3] U. S. Bureau of Mines *Bulletin 344; Technologic Papers Nos. 511, 519, 524, 525, 531, 542, and 543; Industrial and Engineering Chemistry*, Vol. 22, p. 1113 (1930).

[4] Technical Assistant to President, Canada National Research Council, Ottawa, Ont., Canada.

It seems to be very satisfactory for the strongly coking coals, but for those which are near the border line, either at the bottom or the top of the bituminous rank, we ought to have a method rather more sensitive.

MR. W. A. SELVIG.[1]—Our experimental work to date has been concerned principally with evaluating coals having relatively high agglutinating values. For coals having very low agglutinating characteristics, such as those near the boundary line between low rank bituminous coal and subbituminous coal, I believe the test might require some modification. These coals have such low agglutinating properties that it may not be feasible to make tests at different ratios of sand to coal and to plot these results as described in the paper. Probably these slightly agglutinating coals could be carbonized at one ratio of sand to coal, such as the 15 to 1 ratio, and the buttons tested to determine whether they would support a weight of 500 g. without crushing. I would suggest making tests to determine how much coal can be used in the mixture before the carbonized buttons become heterogeneous in internal structure. Tests on some Illinois coals with relatively low agglutinating values show that the carbonized buttons are not homogeneous in structure at the 10 to 1 ratio of sand to coal, and in a few instances abnormal buttons have occurred with these coals even at the 14 to 1 ratio of sand to coal, the abnormality usually consisting of a plane of weakness extending across the button from side to side, which is quite different from abnormality due to the formation of large internal cavities.

MR. N. ISENBERG.[2]—I should like to relate some experiences I have had. I submitted two samples, one of Elkhorn coal No. 3, with volatile matter around 37 per cent, and one of Pocahontas coal, with about 17 per cent volatile matter, to one of the laboratories in our district. They were using the Marshall-Bird procedure, and they reported back a value of about 5000 g. for both the Elkhorn and the Pocahontas coals. They used about a 10 to 1 ratio, and a temperature of about 900 C. I wonder whether these results were due to non-homogeneous buttons.

MR. SELVIG.—Agglutinating value of 5000 g. for Pocahontas and Elkhorn coals are lower than we have obtained on samples of these coals at the 10 to 1 ratio of sand to coal. Buttons non-homogeneous in structure might have occurred in one or both of the coals tested. I do not favor the 10 to 1 ratio as so many coals give buttons abnormal in structure at this ratio.

MR. LATHE.—Mr. Selvig proposes to use several different ratios of sand to coal. Is it not possible that the curves will cross each other, even in the comparatively narrow range used? Would it not be better to standardize on a single ratio of sand to coal?

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[1] Chemist, Pittsburgh Experiment Station, U. S. Bureau of Mines, Pittsburgh, Pa.

[2] Inland Steel Co., East Chicago, Ind.

MR. SELVIG.—I believe it is desirable to know, when testing two coals, whether or not the plotted lines cross at any one ratio of sand to coal as this gives more information regarding the agglutinating characteristics of the coals than could be obtained if the test were made at only one ratio of sand to coal.

MR. FIELDNER.—The U. S. Bureau of Mines hopes to obtain considerable information on the value of these agglutinating tests in practice during the coming year.

The Bureau of Mines efficiency engineer for the various Government heating and power plants is much interested in the possibilities of what this test may show in connection with the caking of coal in hand and stoker fired furnaces. He is observing the performance of various coals on which the agglutinating indices have been determined. At the end of another year we hope to have some data as to whether this test is of any value in predicting the caking of coals in fuel beds.

# RELATIVE MERITS OF FILM AND PAPER FOR INDUSTRIAL X-RAY WORK

BY ANCEL ST. JOHN[1] AND H. R. ISENBURGER[2]

## SYNOPSIS

The requirements of a satisfactory radiograph are discussed. Means and ways are shown how to obtain and maintain a desirable standard. Both film and paper should conform with these requirements. That this is the case was proved in two different ways.

First, an attempt has been made to establish reliable exposure curves for steel. This has been accomplished by building up lead steps of 0.005-in. foil and exposing these test specimens under various conditions. The results on the film negatives have been interpreted by means of a Martens photometer and these densities have been arbitrarily applied to the corresponding paper negatives. Density curves for lead penetration have thus been established and these have been translated into the equivalents for steel.

Then, the correctness of these data was checked by exposures of steel plates ranging from ¼ to 2 in. in thickness. The sensitivity was proved by placing two markers, one 1 per cent and the other 2 per cent of the relative plate thickness, on the surface of each specimen. The relative results on film and paper were evaluated in a table.

The conclusions drawn from these tests are in favor of paper for routine industrial X-ray work.

## RADIOGRAPHIC REQUIREMENTS

The primary purpose of this paper is to compare a satisfactory grade of X-ray film with a satisfactory grade of X-ray paper in so far as speed, convenience and cost are concerned. A secondary purpose is to stimulate the establishment and adoption of standards for minimum sensitivity, acceptable quality and other factors which determine the value of a radiograph as evidence of the internal condition of the object it represents.

A standard of minimum sensitivity is already in use in the radiographic inspection of welded seams in pressure vessels. A marker of material similar to the base plate and having a thickness 2 per cent of the thickness of the plate is placed alongside the weld, and the exposure technique must be such that a hole in this marker is distinguishable on the negative. (A requirement to this effect has been incorporated in the Boiler Code of the American Society of Mechanical Engineers.) To set up further standards may be difficult, but suitable standards or criteria of satisfactory quality in X-ray negatives are desirable from the viewpoint both of X-ray operators and of buyers of equipment for which radiographic examination has been specified. We suggest that an acceptable negative must show at least the following:

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[1] President, St. John X-ray Service Corp., New York City.

[2] Secretary, St. John X-ray Service Corp., New York City.

1. Perceptible shadows through the most opaque portion under examination;
2. Contrast sufficient to disclose a 2-per-cent marker through the thinnest portion under examination;
3. Fair definition of the hole in the 2-per-cent marker; and
4. Unmistakable identification of the region examined.

Provided these requirements are met, the minimum density of the negative corresponding to the most opaque portion of the object, the maximum density corresponding to the most transparent portion, and the general density corresponding to the normal portions may each vary over a considerable range. Certain limiting values of the general density are, however, preferable. Thus, it should not be necessary to examine the negative at a glancing angle because it is too "thin" or to view it against a strong electric light because it is too dense.

Of the four requirements set forth above, the first can be secured by placing a marker of some sort over the most opaque part on the side toward the X-ray tube; the second requires the use of the proper voltage across the X-ray tube; the third is controlled by the relative positions of tube, object and negative; and the fourth is secured by placing a numbered marker at a convenient position in the region covered by the exposure. In many cases a single marker will take care of the first and fourth requirements.

Since the tests described in this paper involve determination of density of X-ray negatives, it is well to recall at this point that this density depends upon the response of the photo-sensitive emulsion to the rays; the technique used in developing the latent image; the characteristics of any fluorescent screens used to intensify the image; the quality and intensity of the X-rays reaching the point; and the duration of the exposure. The quality and intensity of the rays reaching a point of the negative depend in turn upon the quality and intensity of the X-rays themselves (which are functions of the voltage applied to the X-ray tube, the current flowing through it, and the material forming the target in which the rays originate) and upon the composition and quantity of any material traversed by the rays. The mathematical statements for all this are complex and are omitted here, but the relationships may be shown conveniently by suitable graphs.

## DESCRIPTION OF TESTS

The tests described herein were made to compare a satisfactory grade of X-ray film with a satisfactory grade of X-ray paper. The characteristics compared are sensitivity, contrast, convenience in use, and cost. The tests were so carried out as to satisfy the first and second requirements previously mentioned, under conditions conforming to the third and fourth requirements. Four conditions of operation such as might be considered for routine service were used, designated below as conditions *A*, *B*, *C* and *D*. Two conditions (*A* and *C*) are reasonable, while the other two (*B* and *D*) are intentionally unreasonable in order to bring out the effect of unsuitable conditions.

To evaluate the sensitivity and contrast characteristics of film and paper, a test specimen was prepared comprising 16 steps of lead foil each 0.005 in. thick; that is, first step 0.005 in., second step 0.010 in., third step 0.015 in., and so on up to 0.08 in. The steps thus varied by one sixteenth of the maximum thickness. This specimen was used in conditions *A* and *B*. For condition *C*, 14 layers of foil each 0.005 in. thick were added, giving steps from 0.075 to 0.150 in., varying by one thirtieth of the maximum thickness. For condition *D*, 28 layers of the foil were added to the first specimen, giving steps from 0.145 to 0.220 in., varying by one forty-fourth of the maximum thickness. Exposures were made at 4 ma. current with the negative at 24 in. from the source of X-rays, under the following conditions:

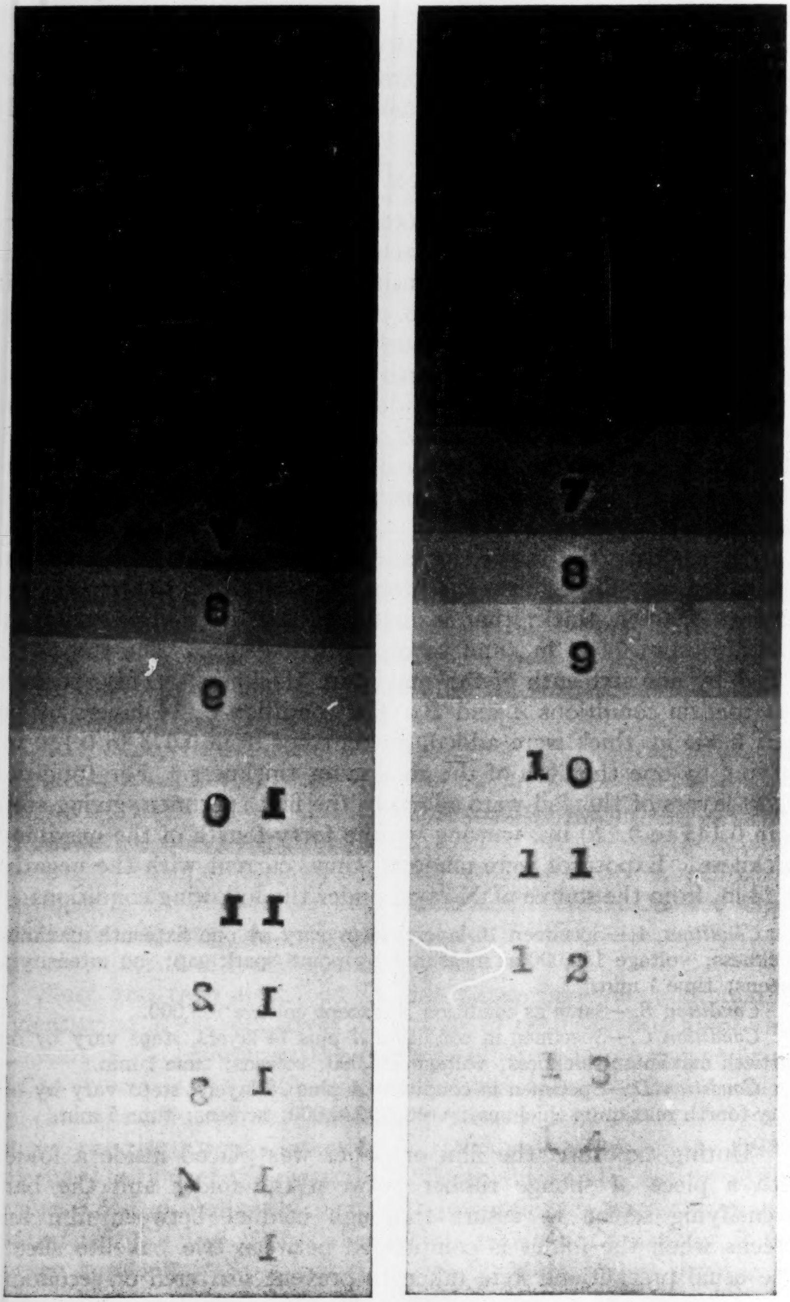
*Condition A.*—Specimen 16 layers, steps vary by one sixteenth maximum thickness; voltage 150,000 as measured by point spark gap; no intensifying screens; time 1 min.

*Condition B.*—Same as condition *A*, except voltage 185,000.

*Condition C.*—Specimen in condition *A* plus 14 layers, steps vary by one thirtieth maximum thickness; voltage 185,000; screens; time 1 min.

*Condition D.*—Specimen in condition *A* plus 28 layers, steps vary by one forty-fourth maximum thickness; voltage 230,000; screens; time 5 min.

During exposure, the film or paper was placed inside a folder, with a piece of sponge rubber between the folder and the back intensifying screen to assure thorough contact between film and screens when the folder is compressed between two bakelite sheets. The usual precautions were taken to prevent scattered or secondary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(a) Film. (b) X-ray paper.

FIG. 1.—Exographs of Lead Step-Test Specimen.

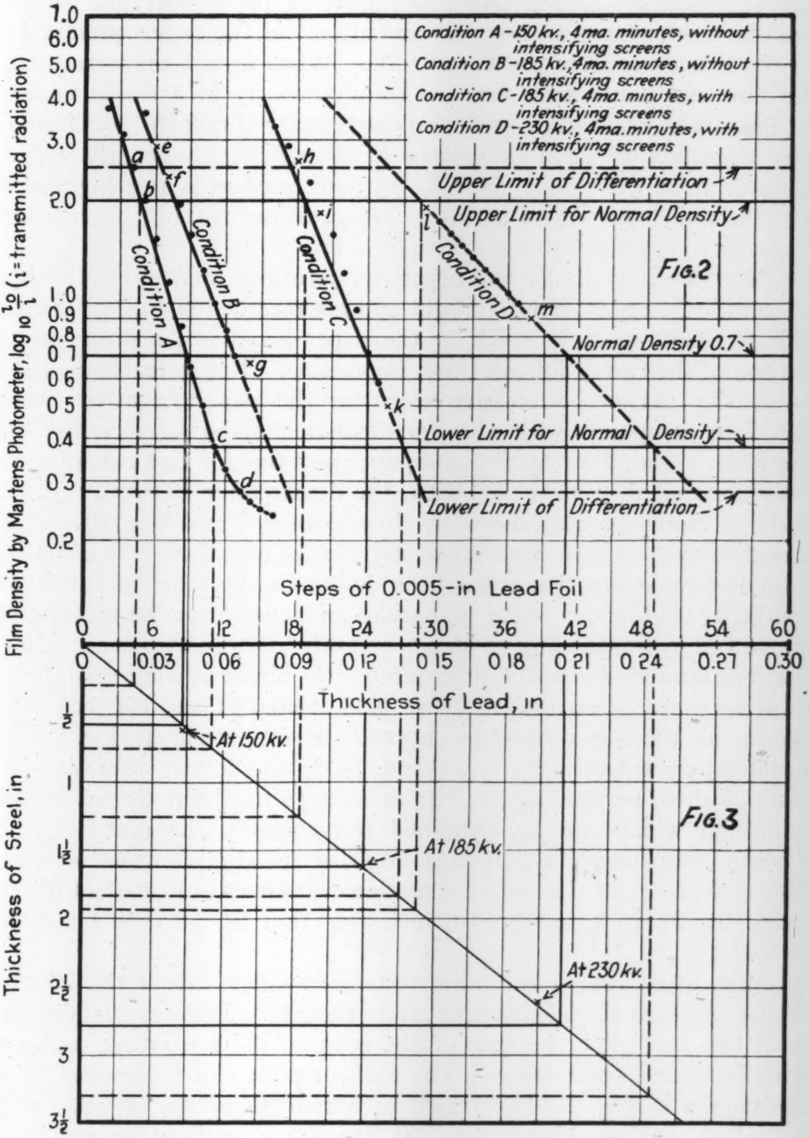

FIG. 2.—Film Density Charts as Determined by Tests.

FIG. 3.—Chart Showing X-ray Equivalence Between Lead and Steel.

X-rays reaching the negative. With X-ray paper, but one screen need be used, placed against the coated face of the paper, preferably on the side away from the X-ray tube. When no screens were used, the film and paper were exposed simultaneously in a single folder; in the other cases they were exposed under duplicate conditions. The negatives of each pair were developed simultaneously for 5 minutes at 65 F. (18 C.), fixed for 10 min., washed in running water for 15 min., and dried at room temperature without forced air circulation, paper requiring 2 hr., film 3 hr.

The densities of the film negatives were measured by a Martens photometer using transmitted light and are recorded in Fig. 2.[1] Since reflected light must be used for the paper negatives, a direct comparison of the two is difficult if not impossible. Recognizable changes in density between successive steps on the paper negatives were therefore compared with the corresponding steps as recorded on the films. The film negative and paper negative for test condition *A* are compared in Fig. 1. In both, the demarcation between steps 3 and 4 is easily recognizable while that between 2 and 3 is not. The film density 2.53 corresponding to step 4 is the maximum which can be examined conveniently and has been designated by *a* on the curve for condition *A*, Fig. 2. Some gradations of density can be recognized above 2.01, corresponding to step 5, and this has been chosen as the upper limit for normal density, designated by *b*. Similarly some gradations of density can be recognized below 0.38 at step 11 and this has been chosen as the lower limit for normal density, designated *c*. The demarcation between steps 13 and 14 is the lowest that can be distinguished, so the density 0.28 at step 13 has been chosen as the minimum which can be examined conveniently, designated by *d*. There is no perceptible difference in either speed or contrast between film and paper for this set of conditions; both record satisfactorily a range from step 4 to step 13, inclusive, that is, 0.020 to 0.065 in. of lead.

The negatives for the other tests are similar, except that at the higher voltages the contrast between successive steps is reduced for both film and paper because of the reduced differentiating capacity with increased penetration. The useful results can be learned from the curves in Fig. 2. The region of normal densities, or what may be called the "working range," at 185,000 v. without screens (test condition *B*) is from step 7 to step 16 or 0.035 to 0.080 in. of lead, while with screens (test condition *C*) it is from step 19 to step 27 or 0.095

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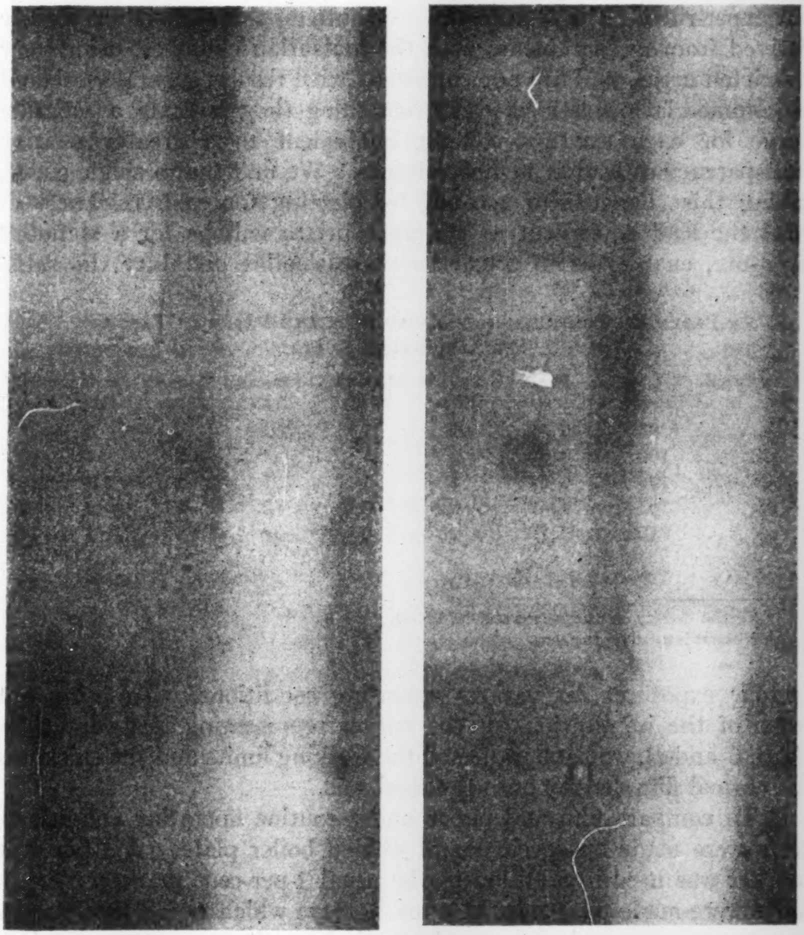
[1] Photographic density is defined as the logarithm to the base 10 of the ratio of the light incident on a photographic deposit to the light transmitted by that deposit.

to 0.135 in. of lead. The thickness for a normal density of 0.7 such as is recommended for general routine is about 0.060 in. of lead without screens and 0.120 in. with screens. This illustrates one advantage of using screens. The minimum densities secured in test conditions *B* and *D* are both greater than the lower working limit 0.38. The voltage was too high in condition *B* and the exposure too long in condition *D*.



(*a*) Film. (*b*) X-ray paper.

FIG. 4.—Exographs of Welded Boiler Plate 1 in. Thick. Note hole in 4-per-cent marker.

To correlate these results on lead, which metal is used chiefly as a protective material because of its high absorptive capacity, with a commercially useful material like steel, we use a graph, Fig. 3, representing the X-ray equivalence between lead and steel, according to our experience. The data in the literature on this subject are mainly derived from measurements with the ionization chamber, using very restricted areas, and are not consistent with results generally secured in commercial operations. For one thing they indicate a separate curve for each exciting voltage, equivalent to a greater relative transparency of lead at higher voltages. We find that a single curve, taking this change into account by plotting for each thickness of steel the lead equivalent at the appropriate voltage for a standard exposure, enables us to predict with reasonable certainty the satisfactory exposures for various operating conditions. To bring out some of the implications of the curves representing test conditions *A*, *B*, *C* and *D*, we have projected the working limits and the thickness for normal film density onto the steel axis.

TABLE I.—COMPARISON OF X-RAY FILM AND PAPER IN TEST OF WELDED BOILER PLATE.

| PLATE THICKNESS, IN. | KILOVOLTS | FILM DENSITY[a] | 1-PER-CENT MARKER | | 2-PER-CENT MARKER | | SCORE | |
|---|---|---|---|---|---|---|---|---|
| | | | FILM | PAPER | FILM | PAPER | FILM | PAPER |
| ¼.......... | 95[b] | 0.56 | 1 | 1 | 2 | 2 | 3 | 3 |
| ⅝.......... | 100 | 0.50 | 2 | 1 | 2 | 2 | 4 | 3 |
| 1.......... | 125 | 0.32 | 1 | 1 | 2 | 2 | 3 | 3 |
| 1½.......... | 165 | 0.43 | 0 | 0 | 2 | 2 | 2 | 2 |
| 2.......... | 230 | 0.79 | 1 | 0 | 2 | 1 | 3 | 1 |
| | | | | | | | 15 | 12 |

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[a] Nearest density on comparison standard films.

[b] Without intensifying screens.

To compare film and paper under routine operating conditions, tests were made on specimens of welded boiler plate. A 1-per-cent marker was used in addition to the usual 2-per-cent marker. Exposures were made for 1 min. at 4 ma. to rays which would surely show the 2-per-cent marker and might disclose the 1-per-cent marker. To get a numerical scale of relative merit a score of 0 was assigned when a marker could not be discerned, 1 when it could be discerned with effort, and 2 when discerned without effort. The results, given in Table I, show that the 2-per-cent marker is readily found in all cases but that the 1-per-cent marker is more often distinguishable on the films than on the papers. Unfortunately the loss of contrast in reproducing such negatives makes it difficult to present illustrations for these tests. Figure 4 compares film and paper for 1-in. plate with a 4-per-cent marker.

## SUMMARY

The tests show that the film and the paper are equivalent in so far as speed is concerned. The paper has a slightly greater range in thickness recorded satisfactorily for the same exposure and consequently has a slightly lower differentiating capacity represented by the score 15 to 12 on a somewhat arbitrary rating. Prints can be made from film and satisfactory photostats from paper, for reproduction purposes. Since only one intensifying screen is required for a paper negative, two duplicate negatives can be made with the paper without modifying the holders. The cost of the paper is less than half that of film, so this can be done with no additional expense. Films must be examined against a uniformly illuminated background; the paper can be viewed in any sort of direct light. The paper dries faster than film. Being coated on one side only, it is less subject to damage in handling.

Hence it appears that with respect to speed and contrast there is little to choose between film and paper; with respect to general convenience the balance is in favor of the paper; and with respect to cost the paper is considerably less expensive than film.

*Acknowledgment.*—The authors are indebted to V. B. Sease, Director of the Redpath Laboratory of the DuPont Film Manufacturing Co., for the density measurements, and to the Metal and Thermit Corp., for furnishing the welded steel plates.

# PLANNING FOR THE COLLECTION OF STANDARDIZATION DATA

By J. R. Townsend[1]

## Synopsis

This paper written from the point of view of the work of the American Society for Testing Materials deals with a method of planning for the collection of standardization data. It is shown that careful planning and much preliminary work are necessary in order to obtain good data. The use of the data depends upon a thorough knowledge of how the data were obtained. Means are discussed whereby bad data may be avoided and useful data obtained. Two engineering investigations leading to standardization by the Society are discussed as illustrations of how this method of planning works out in practice. The fundamental method involves a careful consideration of the problem, and all the factors involved, classification of all the variables as to their relative importance, use of simple but adequate methods of test and by the use of statistics a tool is provided for efficient handling of the data leading to logical conclusions. The method of planning discussed is recommended for use and is explained in its broader aspects as well as in the details of its application to the two specific problems.

One of the important functions of the American Society for Testing Materials is the development of standard specifications and methods of test for materials. The committees responsible for the preparation of these standards collect data and interpret their relation to the problems in hand. In view of the importance of these specifications and methods of test to industry, their preparation requires that the most efficient use be made of all available information.

To tap all available sources of pertinent information, not only must the relevant literature be consulted, but recourse must be had to those familiar with the industrial and economic background of the problem, preferably by including on the committee practical men acquainted with the sources, natural or industrial, of the materials involved, and with their behavior in manufacture and in service. Usually new data are needed, and to plan and conduct the necessary tests the services of experts in theoretical and applied physics and chemistry are required. All these contributors to the investigation can aid in evaluating the significance of the information collected.

The services of experts in statistical methods may be desirable. Since test results and the properties of materials always exhibit some variability, it is necessary to collect sufficient data to determine the extent of this variability. Obviously there is an economic limit to this procedure in any given case. On the other hand, our judgment concerning a physical constant, method of measurement, or degree of variability of a product cannot be based upon a single observation. It is therefore necessary to collect a number of observations. Statistical methods are helpful in extracting from the total information contained in the raw data a few simple functions which express the essential information.

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The data on which standardization is based serve a variety of purposes of scientific, industrial, and economic importance. They serve, for example, in determining the distribution of quality characteristics for materials, in setting economic standards of quality and comparing the relative merits of two or more materials for particular uses, in controlling quality at desired levels, and in predicting what variations in quality may be expected in materials subsequently produced. They aid in discovering the relative distributions of errors of measurements when comparing two or more methods of test, in specifying the precision of standard tests and in setting up economical testing and sampling procedures. Such studies aid in determining relationships between two properties of a material or between direct or indirect measures of physical properties. They may be used to find the physical causes of the behavior of materials under particular service conditions and to reveal the conditions responsible for non-conformance with specification standards.

There are discussed below a number of considerations that are important in planning for the efficient collection of standardization data. These considerations have served as a basis for the successful conduct of several A.S.T.M. projects. They deal not only with the efficient use of data but with the analysis of data in relation to all available knowledge of the material and of the test methods involved. The application of these considerations is illustrated by brief outlines of the course of two actual projects.

## GENERAL CONSIDERATIONS

Considerable judgment is required in establishing quality standards, specification limits and methods of test. The standards selected must be commercial and permit a reasonable variability in manufacture, while affording sufficient control to assure the consumer or user of materials of a satisfactory level of quality. Any raw material or manufactured product will exhibit different levels of quality and different degrees of variability corresponding to the various sources and methods of manufacture from which it is obtained. In setting standards of quality, consideration must be given to the economic background of the material or product both in source and service. Standards selected must, if possible, permit the material to be bought in a competitive market, but exclude faulty or low-grade material. Since most manufacturers constantly improve the quality of their product, provision must be made to admit material of better quality as it becomes available.

The quality desired in a material may be had in several practical ways:

1. By purchasing from one or more suppliers who have an understanding of the needs of the purchaser.
2. By purchasing materials according to established brands.
3. By specifications.

Where large quantities of materials are purchased, specifications are most satisfactory since they permit buying in a competitive market. More important still, specifications assure the purchaser of a uniform level of quality allowing him to plan his fabrication to fit the material with the least possible difficulty in manufacture due to variability. Also, if the producer sells his product to national standards, any purchaser large or small knows what he is paying for.

Specifications may be drawn up in several ways. The most direct method is to specify the quality desired in terms of the basic properties that determine the behavior of the material in service. Such requirements may be called "end point requirements." Sometimes it is not possible to do this economically and the desired quality must be controlled indirectly. Many of the simplest quality characteristics of material cannot be directly measured; for example, smoothness or roughness, tackiness, slipperiness, oiliness, etc. This group may be controlled by observational standards: relative roughness may be judged by comparison with standard surfaces of mild steel which have been grit blasted with a definite size of grit under controlled conditions. Other quality characteristics such as wear resistance, abrasion resistance, freedom from internal strain, etc., cannot be readily measured. Wear or abrasion resistance may be controlled by a specified wear test under mutually agreed conditions, or, as is more frequently done, the wear resistance is controlled by tests of the properties which have been correlated with wear in service tests.

If quality characteristics such as wear resistance, tensile strength and hardness are designated tests $A$, $B$ and $C$, respectively, and if $A$ is highly correlated with $B$ and $C$, $A$ may be controlled by controlling $B$ and $C$. For example, a tensile strength and a hardness test may be specified for a particular metal. The metal meeting such requirements would normally have a definite wear resistance, and it would be incompatible that this resistance should vary greatly. Tensile and hardness tests are frequently used to control some other property in which we are more directly interested from an engineering standpoint, such as formability, bendability, rigidity, etc.

Source identifying requirements may also define the quality of materials. Here again it is recognized that materials of a definite source will fulfill a number of useful purposes within well-established limits. Grade II Chemical Lead bears a source identifying requirement in that it be refined from Southeastern Missouri ores.[1] The process of manufacture may also be specified as in the case of steel specifications where either bessemer or open-hearth steel may be specified.[2]

A 100 per cent inspection test will give the most complete picture of the quality of materials. Such thorough inspection is not necessary for most uses of materials; it is costly, and in the case of destructive or partially destructive tests it cannot be applied. In some instances a test may be destructive or non-destructive depending upon how the part is used. For example, the indentation left by a hardness test may not be objectionable for some parts; yet it would be objectionable on bearing surfaces, and on other parts where appearance is a primary consideration. Where destructive tests must be applied, it is of fundamental importance to devise sampling tests and develop scientific methods of quality control. Methods for economic control of quality of a manufactured product have been published elsewhere.[3]

Other important items must be considered in gathering standardization data for specification use. The supplier must agree to meet the test requirements, should be familiar with the test methods, and have equipment for carrying out the test. The test methods should be widely used and generally recognized wherever possible. The equipment used should be that which a supplier would normally have in his plant. Whereas it would be possible to devise a variety of hardness, ductility, viscosity, and consistency tests, it is essential first to consider carefully whether existing tests are satisfactory. The burden of defending a new test is definitely on the shoulders of its sponsor. With increasing use of new materials, and of old materials in new applications, testing engineers are, however, constantly developing or improving tests and receiving the hearty support of the producers.

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[1] Standard Specifications for Pig Lead (A.S.T.M. Designation: B 29 – 23), 1930 Book of A.S.T.M. Standards, Part I, p. 594.

[2] Standard Specifications for Low-Carbon-Steel Splice Bars (A.S.T.M. Designation: A 3 – 24), 1930 Book of A.S.T.M. Standards, Part I, p. 30.

[3] W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., New York City (1931).

H. F. Dodge, "Statistical Control in Sampling Inspection," *American Machinist*, October 26 and November 9, 1932.

These fundamental considerations in collection of standardization data form a background which must be taken into account if tests are to be planned successfully. Insight and ingenuity must be added in developing test methods; also a thorough comprehension of the material, its sources, and economic background; and skill and boldness in selecting the most important factors for test from a sea of variables.

Below is a list of items to be considered in collection of standardization data or in conducting round-robin tests. Each problem may have its own peculiarities and therefore a single plan will not fit all cases—no chronological order of the various stages of investigation that will suit all conditions. It is believed, however, that this list includes most of the factors that normally occur.

Before giving these considerations it is well to emphasize again that if the data are to be used efficiently and yield the results hoped for, it is necessary that we clearly comprehend the test and have a thorough physical conception of the methods employed and the results obtained.

## ITEMS TO BE CONSIDERED IN THE COLLECTION OF STANDARDIZATION DATA AND IN ROUND-ROBIN TESTS

### *Preliminary*

1. STATEMENT OF THE PROBLEM.

In clearly stating the problem, its object, the particular field to be covered, and the conditions to be considered, the attention is focused on a specific problem rendered in as simple a form as possible and shorn of unnecessary details. The preliminary statement may later be modified by the turn of events in the collection of data.

2. SELECTION OF INVESTIGATION PERSONNEL IF NECESSARY.

The personnel selected should include those who have the most intimate knowledge of the particular field or fields covered by the problem. It is frequently necessary to consult or seek the aid of specialists in particular allied fields. Testing experts and those who have made a study of the means to be employed for the measurements should be consulted and their cooperation solicited. Such experts are frequently referred to as general interests to differentiate them from producers and consumers.

3. ANALYSIS OF THE PROBLEM.

*A.* LIST AND CONSIDER THE POSSIBLE FACTORS THAT MAY INFLUENCE THE FEATURE OF THE MATERIAL OR TEST UNDER INQUIRY, THE CONDITIONS OF USE OF THE MATERIAL, PRODUCT OR TEST.

*B.* COLLECTION AND REVIEW OF ALL PERTINENT PRIOR KNOWLEDGE.

This should include all qualitative and quantitative data, published and unpublished findings from the work of others, regarding

(*a*) physical factors that affect the variable under inquiry,
(*b*) methods of test available,
(*c*) precision of the various tests.

*C.* DIVISION OF THE PROBLEM BY PHYSICAL AND CHEMICAL LAWS INVOLVED.

*D.* SEGREGATION OF THOSE PORTIONS OF THE PROBLEM REQUIRING SEPARATE INVESTIGATION WHERE PRIOR KNOWLEDGE CANNOT BE USED.

This involves the selection of the precise fields to be investigated and consideration of what factors are to be held constant and what factors are to be varied and in what way.

4. SELECTION OF THE TESTING EQUIPMENT.

In selecting testing equipment, consideration must be given to availability, simplicity, ease of operation, precision, and limitations with regard to systematic and personal errors. The error of measurement of the testing equipment should be determined with relation to the overall accuracy required of the test results.

5. SELECTION OF EXACT CONDITIONS UNDER WHICH TESTS ARE TO BE MADE AND EXACT METHOD OF PROCEDURE.

This involves narrowing the field of the problem and the selection of the test conditions and equipment.

6. SELECTION OF METHOD OF CALIBRATION OF TESTING EQUIPMENT TO BE USED BY ONE OR MORE COLLECTORS OF DATA.

This should include also any schedules for checking, calibration, the number of tests that should be made and the tabulation of the data.

7. DETERMINATION OF METHOD OF DRAWING REPRESENTATIVE SAMPLES TO BE MEASURED.

8. STATEMENT OF ACCURACY DESIRED IN FINAL RESULTS FOR EACH SUBGROUP OF TESTS.

This involves the determination of the number of observations required for the desired accuracy.

9. COLLECTION OF PRELIMINARY DATA.

The collection of preliminary data is frequently required to settle some of the above questions which cannot be answered from prior knowledge. See Items Nos. 4, 5, 6, 7 and 8.

10. PREPARATION OF INSTRUCTIONS TO BE FOLLOWED IN THE COLLECTION OF DATA.

In the case of one or more collectors of data and especially in the case of round-robin tests where many collectors of data are involved, instructions should be issued on testing equipment to be used, list of independent conditions for which a group of observations is wanted, number of observations for each condition, specific method of procedure for each condition, method of calibrating and checking testing machines, list of factors which may possibly affect results but which have been presumed to be of minor importance.

### *Collection of Data*

11. TABULATION OF DATA IN SUBGROUPS CORRESPONDING TO THE SEVERAL SETS OF CONDITIONS.

This is an aid to a study of the inter-relationship of all factors involved.

12. OBSERVATION OF TREND OF RESULTS AS INVESTIGATION PROCEEDS.

Watch for unsuspected sources of variation or error as the test results are being collected. A discovery of such effects requires a complete change in the course of the investigation. Careful notes should be taken during the course of the work of factors which seem to affect the results and particularly those that were originally considered unimportant.

13. PRESENTATION OF THE DATA IN A FORM LEADING TO LOGICAL CONCLUSIONS.

This involves the statement of the data in its simplest form[1] leading to logical conclusions where the degree of variability and accuracy obtained form a logical sequence to the statement of object and limitations of the problem.

The above are items which should be considered in planning for the collection of standardization data. The preliminary stage of the work should include a long view of the problem before collecting the data. As the data are collected, the relation of the individual test results to the total data should be watched for unforeseen variability and unsuspected factors. The preliminary factors discussed above outline the initial plan of procedure, but we should be ever alert to change the course of the investigation if events show that we have been misguided.

Relative to the problem of the material involved and a thorough familiarity with the history, prior knowledge and economic background of the material involved are essential foundations upon which plans for collection of data must be based. There is no known way of making bad data good and test results are no better than the care and thought given to their planning and execution. Granting we are thoroughly familiar with all of the factors involved in the test and the material, we may then efficiently collect and group the data by the use of statistics and reach logical conclusions in the most direct manner.

The various steps may not occur here exactly in the order in which they appear in a particular problem. They are also interrelated but they offer in substance the items which have proved valuable in practice.

This procedure, as a whole, is naturally based upon the "scientific method" involving observations, hypotheses and experimental verifications. The investigator must construct from his observations a mental model of the phenomenon and test its consistency with itself and its concordance with results of further experiments. The resulting hypotheses are not something fixed but are subject to change if in any stage in the investigation the observed data are definitely inconsistent therewith.

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[1] See Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I (1933); also published in pamphlet form.

The observations or measurements of scientific research are necessarily precise since they should determine relationships very accurately. The usual method is to control all variables and to measure the effects produced by a single variable, making all measurements in terms of mass, length, and time or in units that may be referred to these fundamental units. While this scheme of carefully measuring the effect of each variable is a precise method of study, the materials and structures employed in engineering are often so complex that it is not economically possible or desirable to determine the effect of each variable separately and show their exact interdependence. Even this information would not be useful in many instances since we would still have to determine the relative effect of many variables. It is necessary, therefore, in carrying out an investigation to take a bold step in planning the work and to subordinate the effects of many variables in the problem. This requires a careful consideration based on preliminary experiments and on experience, to decide which variables may be subordinated or ignored in the tests. In fact skillful use of this method is really the basis of many tests that are used in A.S.T.M. specifications to control the quality of materials.

Thus far we have considered the collection of good data. To be sure that our data are good, we must avoid certain common pitfalls which result in bad data. These various pitfalls may be summarized briefly as follows:

*Over Simplification of a Test in Order to Obtain Reproducibility:*

By modifying the test condition an investigator may get results which have a higher degree of reproducibility, and this is generally considered highly satisfactory. One must be especially careful, however, to avoid over simplification that would eliminate extremely important variables affecting the fundamental objective of the work. Several of the steps in an investigation may be eliminated in instances where only rough comparisons are desired; in other instances we may not know beforehand what degree of accuracy is required in our measurements. It is necessary, therefore, to gather experience by using the best means at hand. As a corollary, however, it is obviously fruitless to make observations with apparatus not sufficiently precise and equally wasteful to make observations with apparatus too precise.

### *Presupposition of the End Result:*

It has been said somewhat paradoxically that ideas are the negation of thinking. It is useful to have a tentative hypothesis as to the cause or effect of engineering results but this hypothesis should be subject to verification as the test proceeds. In other words, the test should be designed to give the phenomenon a fair chance. If we do not do this we may reach misleading results, that is, arrive at a conclusion that is merely a wish-fulfillment.

### *Lack of Flexibility of Program:*

As our tests proceed it is well to keep continually their trend before us. By so doing it is possible to limit the amount of work required, to extend the experimental program, and to discover other factors that may be affecting the result. This last is tremendously important since it determines the whole course and value of the measurements. Much can also be observed by the behavior of the test: do accidental variations in technique or handling seem to affect the result; is the degree of variability greater than expected; do other factors seem more important? By keeping such vigilance over the work one may make discoveries of entirely new phenomena. Statistical methods will aid by indicating the degree of such variability, the significance of the results, whether they are due to a system of chance causes, etc.; but these are only tests, and the cause of the variability must be eternally watched for and recognized as having a physical significance.

## EXAMPLES OF PLANNING FOR COLLECTION OF STANDARDIZATION DATA

### *Determination of Requirements for Non-Ferrous Sheet Metal:*

The data from this investigation on requirements for non-ferrous sheet metal led to standard tests and limits for the control of non-ferrous sheet metal. A complete report of this work was given in three previous papers.[1] On the basis of the standardization data furnished by these papers, by the commentators, and by the members of Committee B-5 on Copper and Copper Alloys, Specifications for Sheet High Brass (A.S.T.M. Designation: B 36 – 27) were drawn up. For the details of this work the reader is referred to the original papers. There are reviewed herewith the general methods employed:

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[1] H. N. Van Deusen, L. I. Shaw and C. H. Davis, "Physical Properties and Methods of Test for Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 173 (1927); J. R. Townsend, W. A. Straw and C. H. Davis, "Physical Properties and Methods of Test for Some Sheet Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 381 (1929); C. H. Davis and E. L. Munson, "Hardness Relationships and Physical Properties of Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 422 (1929).

1. All available methods of test for hardness, tensile strength, ductility, and reversed bending were investigated by correlating them to the tension test. This was done since it was considered that the tension test was basic and the others might be used for inspection or character-defining purposes. Ductility, while useful for qualitative study, was considered unreliable since it varies to a fairly large degree with thickness of the metal, grain size, surface finish, etc. In fact a different set of conditions appeared for each thickness of metal. This was also found to be true of bending but to a smaller degree. These tests were, therefore, dropped because of this variability; and, in the case of bending, for the additional reason that bend testing machines of a suitable character were not generally available. The various hardness tests tried, such as Scleroscope, Brinell, Meyers hardness constant, and Rockwell hardness, showed various facts. The Scleroscope was affected by many variables and, most important, the various Scleroscope machines failed to check each other; the Brinell test was unsuitable for thin sheet metal, the Meyers hardness test, although most satisfactory from a scientific standpoint, could not be used because the equipment was not generally available. The Rockwell hardness test was finally selected for further study since it combined availability, good correlation with tensile strength, good comparison between machines, and was capable of sufficient precision in measuring the hardness of thin sheet metal. Considerable study was given to the comparison of Scleroscope hardness with Rockwell hardness since the former was in general use at that time. Figure 1 shows the divergence between the supplier's readings and the consumer's for the Scleroscope and the Rockwell machines, respectively.

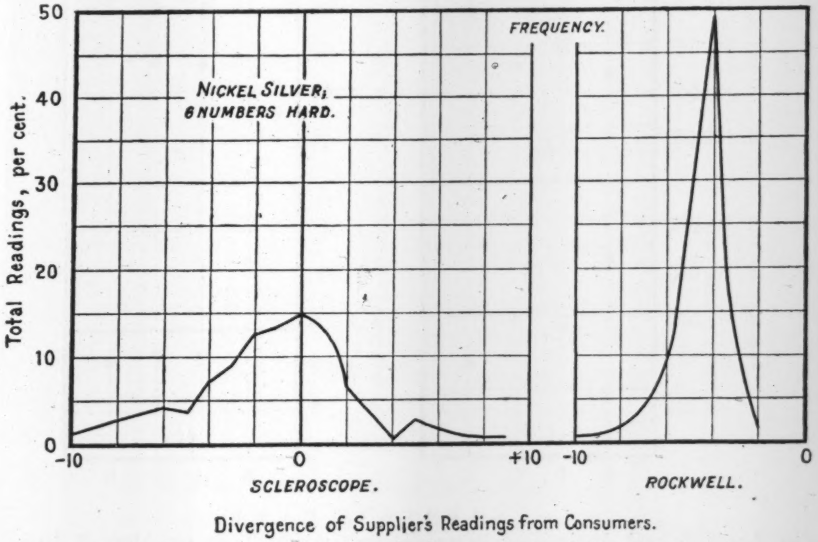

FIG. 1.—Typical Results Showing Frequency of Occurrence of Degrees of Difference Between Two Machines.

2. Preliminary round-robin tests were then made using the same material tested by a number of operators on a number of Rockwell hardness machines. This led to the selection of the proper hardness scale, and control of the variables of the test by the agreement upon and the adoption of a standard procedure.[1]

3. Meanwhile it was seen that the tension test required careful agreement in method to minimize the effect of the variables involved. This agreement was reached and it was found by a statistical study that uniformly prepared specimens of the same material tested by various laboratories gave no significant variation greater than that which could be left to chance. The variables considered were the testing procedure of the various laboratories and the method of milling of the specimens. Figure 2 gives the results of a series of such tests on nickel silver sheet.

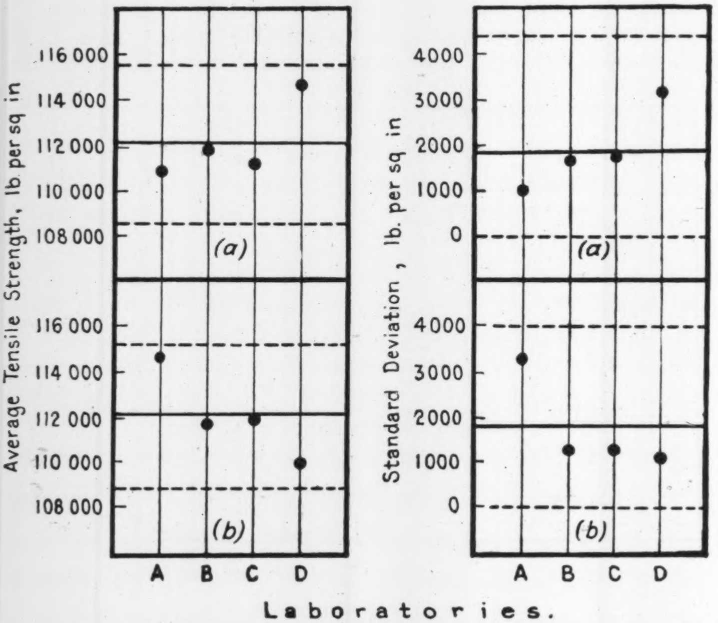

FIG. 2.—Statistical Analysis of Round-Robin Tension Tests on Alloy B Nickel Silver.

(*a*) Points are average of all specimens milled at laboratories indicated, and tested by the four laboratories.

(*b*) Points are average of all specimens tested at laboratories indicated, and milled at the several laboratories.

4. The variables affecting the metal were next considered. These were determined to be chemical composition, annealing temperature as affecting grain size, degree of cold work, tensile strength and hardness. The effect of these variables was determined in the following way. The chemical composition

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[1] Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8 – 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 945 (1932); also 1932 Book of A.S.T.M. Tentative Standards, p. 1077.

limits were those selected on the basis of what could be reasonably expected in a commercial mill. The chemical methods were agreed upon and considerable practical experience with these methods furnished the basis of the limits. The annealing temperature range of the annealing furnaces was known, the resultant effect of annealing on grain size was carefully determined, and representative grain size micrographs were standardized. The variation in tensile strength due to previous annealing temperature range was carefully determined at the mill on a large number of samples.

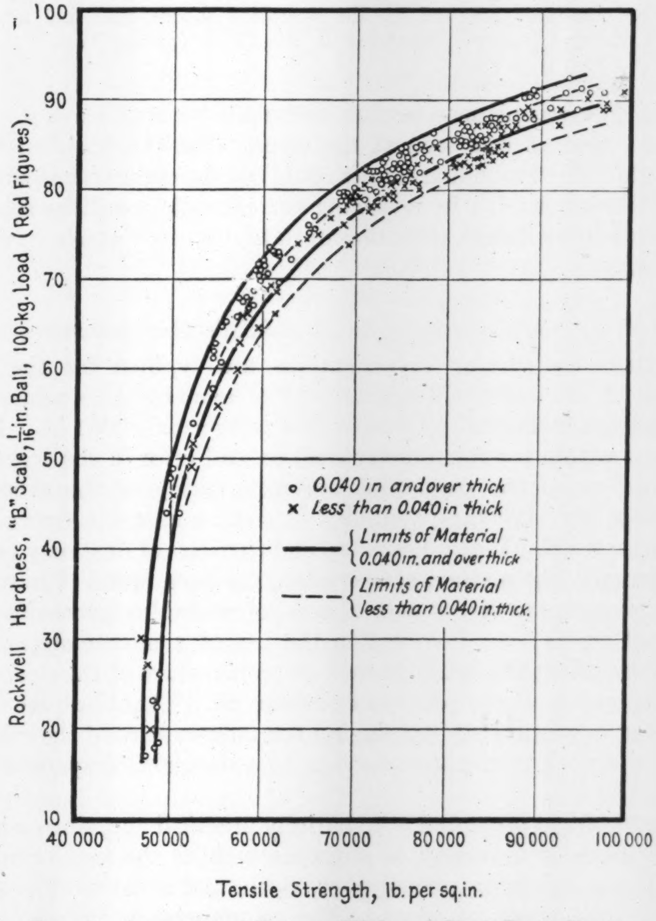

FIG. 3.—Accumulated Data on High and Clock-Brass Sheet.

5. The preliminary stage of the work being completed, the data from the final round-robin tests were then collected. Rockwell hardness readings were compared with tensile strength tests on several rolling series where all grades, tempers and thicknesses of sheet brass were tested. Correlations between

Rockwell hardness and tensile strength limits were drawn up for all tempers and thicknesses of sheet metal based on annealing range and chemical composition.

6. These limits were checked against a large number of orders of commercially manufactured material. It was found that the limits were satisfactory both to the producer and the consumer. A statistical study of the experience data is given in Fig. 3.

7. The standards adopted by the operating laboratories and finally by the A.S.T.M. were Rockwell Hardness E 18 - 32 T, Tension Test E 8 - 32 T, Sheet High Brass B 36 - 27.

The work was planned so that it would be susceptible of statistical treatment since it was seen at the outset that the problem was so complicated that individual results could not show the true significance of the facts involved. In reviewing the example it will be seen that most of the steps in the collection of data discussed above have been carried out.

*Investigation of the Accuracy of the Machinist's Micrometer:*

Certain engineering investigations are typified by the determination of the error of measurement of a machinist's micrometer. These micrometers read by means of a vernier to 0.0001 in. Due to variations within the micrometer such as lead error of the screw, lack of parallelism of the contacting surfaces, spring of the arbor, and calibration, we have the possibility of certain inherent errors in the micrometer itself. Wear, corrosion, and particles of dust may produce other errors. The method of operation, pressure, speed of movement of the micrometer screw and a host of variables generally called "personal errors" are involved in the use of a micrometer. Other factors also affect the readings such as temperature of the micrometer and temperature of the parts to be measured. These last are usually eliminated or minimized by bringing the temperature of the micrometer and the work to the same level or to a standard temperature such as 68 F. (20 C.).

The problem presented is an entirely practical one, since micrometers are used for a variety of purposes such as the measurement of the thickness of paper, phenol fiber, cloth, and sheet metal; and for checking dimensions of parts. The micrometer is called for in a number of A.S.T.M. specifications.

It was found necessary to collect considerable data, and sufficient use of these data required the application of statistical methods. To reduce the problem to practical limits, ten operators and ten micrometers were selected and each operator used each micrometer on 7 Hoke gage blocks. Since micrometers in use are of two kinds—

those with ratchets designed to control torque, and hence pressure; and those without ratchets that are operated by "feel,"—it was necessary to make the tests by both methods. Since the advantage of the ratchet is only obtained by use of a definite technique of operation, two sets of measurements were made. Prior to the taking of the first set no instructions were given. Later a new set of measurements was made to definite instructions. All of the measurements were made at 68 F. ± 1 F. The gage blocks used were accurate to ±0.000005 in. as calibrated by the U. S. Bureau of Standards. The operators could be divided equally into two groups, (*A*) skilled operators and (*B*) unskilled operators. Data were therefore collected covering the following methods of using the micrometer:

1. Use of the micrometer by the operator without specific instructions as to method of procedure employing
    (*a*) the method of "feel,"
    (*b*) the use of ratchet.
2. Use of the micrometer employing ratchet with definite instructions
    (*a*) by a group of skilled operators,
    (*b*) by a group of unskilled operators.

The results finally obtained illustrate at once the value of statistical methodology in analyzing data and the importance of engineering knowledge of the problem in its interpretation. The analysis of the data showed it to have been collected under controlled conditions, and the estimates finally obtained of the various component errors complied with all requirements of consistency with the observed variability. The component errors that could be distinguished were instrumental and personal. The former were trivial compared with the latter, provided the zero adjustment was properly made. The personal error was the same for the method of feel as for the ratchet method when a definite technique was used for the latter. The personal error depended on the individual operator, and in each case involved a constant and a variable component. The former could be largely eliminated by requiring the operator to determine his individual average zero correction, and it was concluded that the remaining variable personal error was essentially the only error under proper conditions of actual use. The general level of this error for average operators gave the accuracy of properly employed micrometers as 0.0002 in.

At the same time, engineering knowledge of the instrument and its use indicated that both types of personal error corresponded to variations in torque applied to the micrometer spindle. From this it followed that the variability observed in these tests, which were made on steel blocks, might be less than that found in similar measurements on more compressible materials or those having rougher surfaces. Additional work has, therefore, been carried out on other types of materials and further studies along this line are still in progress.

TABLE I.—SUMMARY OF TYPICAL RESULTS OF MICROMETER MEASUREMENTS ON A SERIES OF GAGE BLOCKS.

| | Gage Block, in. | Operator 1 | | Operator 2 | | Operator 3 | | Operator 4 | |
|---|---|---|---|---|---|---|---|---|---|
| | | $\overline{X}$, in. | $\sigma$, in. | $\overline{X}$, in. | $\sigma$, in. | $\overline{X}$, in. | $\sigma$, in. | $\overline{X}$, in. | $\sigma$, in. |
| | | MICROMETER No. 1 | | | | | | | |
| By Ratchet..... | 0.0000 | −0.0001 | 0.000000 | −0.00010 | 0.000000 | 0.00010 | 0.000000 | 0.000000 | 0.000000 |
| | 0.0100 | 0.01002 | 0.000040 | 0.00990 | 0.000000 | 0.01000 | 0.000000 | 0.01008 | 0.000040 |
| | 0.0500 | 0.04998 | 0.000040 | 0.05000 | 0.000000 | 0.05000 | 0.000000 | 0.05008 | 0.000040 |
| | 0.1000 | 0.09990 | 0.000000 | 0.09998 | 0.000040 | 0.10000 | 0.000000 | 0.10000 | 0.000000 |
| | 0.2500 | 0.24996 | 0.000049 | 0.24990 | 0.000000 | 0.25000 | 0.000000 | 0.25004 | 0.000049 |
| | 0.5000 | 0.49990 | 0.000000 | 0.49990 | 0.000000 | 0.50000 | 0.000000 | 0.50000 | 0.000000 |
| | 1.0000 | 1.00000 | 0.000000 | 0.99990 | 0.000000 | 1.00000 | 0.000000 | 1.00002 | 0.000040 |
| By Feel......... | 0.0000 | −0.00012 | 0.000040 | 0.00004 | 0.000049 | 0.00000 | 0.000000 | 0.00000 | 0.000000 |
| | 0.0100 | 0.00990 | 0.000000 | 0.01004 | 0.000049 | 0.01000 | 0.000000 | 0.01006 | 0.000049 |
| | 0.0500 | 0.04990 | 0.000000 | 0.05000 | 0.000000 | 0.05000 | 0.000000 | 0.05006 | 0.000049 |
| | 0.1000 | 0.09990 | 0.000000 | 0.09996 | 0.000049 | 0.10000 | 0.000000 | 0.10010 | 0.000000 |
| | 0.2500 | 0.24990 | 0.000000 | 0.25000 | 0.000000 | 0.25000 | 0.000000 | 0.25010 | 0.000000 |
| | 0.5000 | 0.49986 | 0.000049 | 0.50000 | 0.000000 | 0.49990 | 0.000000 | 0.50010 | 0.000000 |
| | 1.0000 | 0.99990 | 0.000000 | 1.00000 | 0.000000 | 1.00000 | 0.000000 | 1.00010 | 0.000000 |
| | | MICROMETER No. 2 | | | | | | | |
| By Ratchet..... | 0.0000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 |
| | 0.0100 | 0.01000 | 0.000000 | 0.01012 | 0.000040 | 0.01000 | 0.000000 | 0.01010 | 0.000000 |
| | 0.0500 | 0.05008 | 0.000040 | 0.05010 | 0.000000 | 0.05000 | 0.000000 | 0.05010 | 0.000000 |
| | 0.1000 | 0.10010 | 0.000000 | 0.10010 | 0.000000 | 0.10002 | 0.000040 | 0.10010 | 0.000000 |
| | 0.2500 | 0.25010 | 0.000000 | 0.25010 | 0.000000 | 0.25008 | 0.000040 | 0.25010 | 0.000000 |
| | 0.5000 | 0.50010 | 0.000000 | 0.50010 | 0.000000 | 0.50010 | 0.000000 | 0.50014 | 0.000049 |
| | 1.0000 | 1.00008 | 0.000040 | 1.00010 | 0.000000 | 1.00010 | 0.000000 | 1.00020 | 0.000000 |
| By Feel......... | 0.0000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 | 0.00000 | 0.000000 |
| | 0.0100 | 0.00996 | 0.000049 | 0.01004 | 0.000049 | 0.01000 | 0.000000 | 0.01010 | 0.000000 |
| | 0.0500 | 0.05000 | 0.000000 | 0.05010 | 0.000000 | 0.05000 | 0.000000 | 0.05014 | 0.000049 |
| | 0.1000 | 0.10000 | 0.000000 | 0.10010 | 0.000000 | 0.10002 | 0.000024 | 0.10016 | 0.000049 |
| | 0.2500 | 0.25000 | 0.000000 | 0.25006 | 0.000049 | 0.25009 | 0.000020 | 0.25016 | 0.000049 |
| | 0.5000 | 0.50002 | 0.000040 | 0.50008 | 0.000040 | 0.50010 | 0.000000 | 0.50014 | 0.000049 |
| | 1.0000 | 1.00000 | 0.000000 | 1.00010 | 0.000000 | 1.00001 | 0.000020 | 1.00018 | 0.000040 |

These results and data were used to establish the use of the micrometer for measuring the thickness of tensile specimens and specimens of insulating materials in A.S.T.M. specifications.

Table I shows a summary of essential information on typical results of micrometer measurements on a series of gage blocks, by four operators, using two micrometers and by two methods.

## CONCLUSION

Some of the various considerations that must be taken into account when planning standardization data have been reviewed.

The methods involved in planning two engineering investigations have been illustrated by examples within the scope of A.S.T.M. work, that is preparation of limits for a material specification, and the determination of the error of measurement of an instrument subject to manipulative control. It will be seen that the fundamental method is the same; a careful consideration of the problem and all the factors involved, classification of variables as to their relative importance, use of simple but adequate methods of test, and the use of statistical methods as outlined in the Manual on Presentation of Data[1] to make efficient use of the data available. The considerations that are important in solving problems involving collection of standardization data, and which have been in successful use, are presented and recommended.

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I (1933); also published in pamphlet form.